

# BRITISH CHEMICAL ABSTRACTS

## B.—APPLIED CHEMISTRY

NOV. 24 and DEC. 1, 1933.\*

### I.—GENERAL; PLANT; MACHINERY.

**Rotary kiln heat balance.** W. T. HOWE (Cement, 1933, 6, 289—296).—The method of calculating a heat balance is based on the fact that the heat vals. per lb. of the various gases and clinker show only a negligible variation from a rectilinear relationship when plotted against the temp. or other factors involved. From results obtained from 24 kilns a general equation represented with fair accuracy the relations between coal consumption and kiln dimensions, added internal surface, clinker output, and slurry moisture. C. A. K.

**Increase of heat transfer through radiation by increasing the velocity of the circulating fluid.** N. NYURENBERG (Groz. Neft., 1932, 2, No. 7—8, 72—76).

**Thermal conductivity of various insulators at room temperature.** C. D. NIVEN (Canad. J. Res., 1933, 9, 146—152).—The thermal conductivities ( $C$ ) of many insulators used in building are recorded. In general, the data lie round a single  $C$ -density curve, but with considerable scatter, and  $C$  increases more rapidly with  $d$  for high densities than for low.  $C$  for animal is  $<$  for vegetable fibres. A. G.

**Emission of dust and grit from power-station chimneys in Holland.** A. J. TER LINDEN (Engineering, 1933, 136, 279—282).—Investigations by the Amsterdam Grit Commission indicated that complaints are due to coarse grit (retained by No. 300 sieve) which is deposited in measurable quantities over an area of approx. 2 miles radius from chimneys 130—200 ft. high; fine grit is carried farther away and causes little trouble. Stations using pulverised fuel emit high proportions of grit, entailing loss of unconsumed fuel; a high-efficiency wet-type catcher should therefore be used. Means are recommended for the regular determination of the grit passing up the stack. C. J.

**Studies in agitation. II. Sand concentration as function of sand size and agitator speed.** A. McL. WHITE and S. D. SUMERFORD (Ind. Eng. Chem., 1933, 25, 1025—1027; cf. B., 1932, 1059).—It is suggested that the agitator ( $A$ ) speed at which max. concn. of the sand suspension is obtained may be used as a criterion of intensity of  $A$ . E. S. H.

**Industrial microscopy. I. Mounting technique for fine powders.** C. H. BUTCHER (Ind. Chem., 1933, 9, 296—297).—Methods of mounting powders on slides are given. D. K. M.

**Calculation of the design of a rotary filter for systematic washing.** R. PALANTE (Chim. et Ind., 1933, 30, 255—276).—For the design of a filter inform-

ation is required as to (1) the solid-liquid ratio in the material to be filtered, (2) thickness of the cake, (3) type of filter-cloth. The solid must not exceed a given max. for good distribution. The vol. and no. of washes and the vac. applied can be calc., as also can the filter dimensions and speed of rotation. The washing curve, which records the fall in concn. of the liquor leaving the filter during washing, must be determined experimentally, the end-point, or permitted loss, being fixed as required. The curve shows a slight initial rise due to  $H_2O$  retained in the filter from a previous treatment. The problem of the no. and vol. of washes is worked out graphically. The liquor passing the filter varies with the sq. root of the suction applied, and, further, the cake becomes less permeable. Again, the temp. places a limit on the possible vac. if boiling is to be avoided. The optimum vac. is given by the washing curve. It rarely exceeds 40—50 cm. Hg. The "dead zone" of the filter which serves for the removal of cake may be taken as 45—55°. The time periods required for given yields in the remainder of the circle are given by trials with an experimental cake, and the necessary surface follows. The no. of cells is fixed by a compromise between the increased efficiency caused by increasing no. and the greater constructional cost. In running, vol. variations due to accidental causes must be compensated by adjusting distributing partitions. Washing is controlled by varying the cake thickness. It is most important to maintain the solid-liquid ratio const. The only experimental apparatus necessary for determining the filtration characteristics mentioned above is a Buchner filter connected to measuring receivers, to vac., and to compressed air. Methods are described in detail. C. I.

**Conservation of filter-press cloth.** M. O. CHARMANDARIAN and L. I. SIVOPLIAS (Ukrain. Chem. J., 1933, 8, 125—126).—The canvas is immersed in boiling 4%  $Na_2CO_3$  during 90 min., and then in ammoniacal  $CuCO_3$  solution during 2 hr. R. T.

**Heating fluids by direct contact.** C. S. DARLING (Ind. Chem., 1933, 9, 305—307).—When heating liquids by direct contact with steam the latter should be taken from the top of the pipe-line. Nozzles and pipes through which steam enters the liquid should be placed centrally in the tank and at some distance from the bottom. Steam should be introduced in as many streams as possible, with flat cross-section. The use of a Sylphon thermostatic mixer and a "Dumbflow"  $H_2O$  heater is indicated. D. K. M.

**Contact equipment for extraction and reaction in two-phase liquid systems.** T. G. HUNTER and

\* The remainder of this set of Abstracts will appear in next week's issue.



A. W. NASH (Ind. Chem., 1933, 9, 245—248, 263—266, 313—316).—The theory of the extraction of a substance from solution with an immiscible solvent is discussed and illustrated. The film concept is applied to the transfer of mass between two fluids in motion. Various types of mechanical mixers, jet and column contactors are discussed and illustrated. D. K. M.

**Rectification in packed columns.** M. WEIMANN (Chem. Fabr., 1933, 6, 411—413).—The relation between vapour concn. and depth of seal in the distillation of a mixture of EtOH and H<sub>2</sub>O in a column (A) packed with Raschig rings was determined. There is an optimum steam velocity for a given reflux depending on the packing and the diam. of A. Researches on heat exchange show the relation to the height with varying steam velocity and reflux; in each case optima exist. Distribution in such a tower is not equal, an excess of liquid passing near the outer walls of A; this effect is increased with increasing diam. of A and of the packing rings. The latter should be  $\frac{1}{2} - \frac{1}{5}$  of the diam. of A. A dished type of tray is described to counterbalance the tendency of the liquid to follow the walls. The pressure loss varies with the steam velocity, type of packing, etc. C. I.

**Evaporation by submerged combustion. I. Experimental equipment.** K. A. KOBE, F. H. CONRAD, and E. W. JACKSON (Ind. Eng. Chem., 1933, 25, 984—987).—Burner apparatus consisting of a mixing chamber into which air and gas are introduced by Cu tubes with side drillings, a velocity tube, and a combustion chamber containing a combustion tube of fused SiO<sub>2</sub> which is surrounded with insulating material is described. From this the combustion gases leave through a grid. The burner is removed from the evaporator to light it. When the SiO<sub>2</sub> tube reaches incandescence it is ready to be submerged. Trials with H<sub>2</sub>O show thermal efficiencies of 85—90%. The fused SiO<sub>2</sub> is not a satisfactory refractory as the wall temp. is 1400—1700°. This method of evaporation is likely to be commercially successful only where difficulties arise through corrosion or sludge formation. C. I.

**De-aëration of boiler-feed waters. Apparatus for determining dissolved oxygen in water.** G. DAMANY (Chim. et Ind., 1933, 29, Spec. No., 268—274).—A simple, portable apparatus is described for removing the gas from samples of H<sub>2</sub>O under barometric vac. and for determining the O<sub>2</sub> content by combustion with H<sub>2</sub>. J. W. S.

**Conditions under which gas and liquid mixtures rise.** J. VERSLUYS (J. Inst. Petroleum Tech., 1933, 19, 694—700).—If a mixture of liquid (I) and gas rising in a tube contains > 50 vol.-% of (I) the foam is stable; if < 50 vol.-% the mist is stable. Mixtures containing 26—50 vol.-% of (I) can form a foam, but it is readily converted into mist; similarly mist conditions with 50—76 vol.-% of (I) are unstable. D. K. M.

**Principles of gaslift.** J. VERSLUYS (J. Inst. Petroleum Tech., 1933, 19, 687—693).—Theoretical. D. K. M.

**Problems of technical reactions. I. Catalytic hydrogenation.** O. SCHMIDT (Z. Elektrochem., 1933, 39, 824—837).—A review.

**Höppler viscosimeter.** C. KAISER (Petroleum, 1933, 29; Motorenbezt., 6, No. 9, 5—6).—The instrument is suitable for use with liquids of viscosities between 0.5 and 10<sup>5</sup> centipoises, the error throughout the whole range being  $\pm 0.27\%$ ; between 10 and 600 centipoises the error is  $\pm 0.1\%$ . Results obtained with 3 different instruments (4 observers) agreed to within 0.3%. Typical results are given. H. F. G.

**Industrial applications of the polarographic method.** J. HEYROVSKÝ (Chim. et Ind., 1933, 29, Spec. No., 204—210).—The polarographic method is described and some of its advantages and industrial applications are discussed. J. W. S.

**[Determining] rate of dissolution.**—See II. **Steels for superheater tubes.** **High-pressure boiler corrosion.** **Thermal conductivity of metals.**—See X. **Radiant energy and paint.** **Determining degree of dispersion of finely-divided substances.**—See XIII.

See also A., Oct., 1026, **Freezing gases at low temp.** **Extremely low temp.** 1027, **Electric sieve.** **Ultra-filtration.**

## PATENTS.

**Control of combustion in metallurgical, boiler, and other furnaces.** BRIT. "REMA" MANUFACTURING CO., LTD., and P. HOWDEN (B.P. 398,200, 8.3.32).—A pulverised-fuel furnace is operated with a const. wt. of primary air (which passes through the pulveriser), and the secondary air is adjusted to the rate of fuel feed so that the total air is in const. proportion to the fuel, the regulation of the secondary air being effected by devices situated before the preheaters or regenerators, i.e., at a point where the temp. of the air is substantially const. B. M. V.

**Catalytic apparatus.** I. HECHENBLEIKNER, ASSR. to CHEM. CONSTRUCTION CORP. (U.S.P. 1,894,768, 17.1.33. Appl., 3.4.29).—A catalyst mass is caused to have a rising temp. gradient in the earlier parts and falling temp. in the later parts by means of concentric return tubes carrying an independent cooling fluid which at the heat-exchanging surface flows in the same direction as the gases. B. M. V.

**Heat-energy transfer medium.** H. H. DOW, ASSR. to DOW CHEM. CO. (U.S.P. 1,893,051, 3.1.33. Appl., 30.10.26).—The medium comprises Ph<sub>2</sub>O with  $\geq 25\%$  of C<sub>10</sub>H<sub>8</sub>, pyrene, or *p*-hydroxydiphenyl. B. M. V.

**Lids for pressure-resisting vessels in which fluids are stored or generated under pressure.** C. S. HALL, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 397,899, 3.2.32).—A closure having an expanding ring is operated by a combination of bayonet and screw. B. M. V.

**Crushing or reducing machines.** EISEN- & STAHLWERKE OEHLE & CO. A.-G. (B.P. 398,121, 29.3.33. Switz., 21.9.32).—The apparatus comprises two or more knives rotating in a conical casing (A) provided with fixed knives, the lower part (apex) of A being cut away to permit free outflow of material. B. M. V.

**Crushing machine.** E. B. SYMONS, ASSR. to NORDBERG MANUFACTURING CO. (U.S.P. 1,894,601, 17.1.33. Appl.,



20.2.29).—In a gyratory cone crusher, means for permitting the bowl to yield to uncrushable pieces are described.

B. M. V.

**Ball or tube mills.** OFENBAU-GES. M.B.H., and H. BANGERT (B.P. 398,000, 23.6.32).—In a dryer and ball mill combined in one shell, the gas which bears away the ground material is returned to the drying chamber (with any oversize separated in external sifters) but through an axial pipe extending into the drying part so that the old does not mix with the fresh drying gas and material until the latter has been dried. B. M. V.

**Apparatus for mechanical separation of materials.** J. A. COOK (B.P. 398,210, 5.2.32).—Material, part (A) of which tends to roll and the remainder (B) to slide, *e.g.*, coal and stone, preferably graded, is passed over a no. of inclined cascades of which the surface friction is increased by, *e.g.*, perforations, each cascade being formed with one or more intermediate narrow gaps to collect B and with wider gaps at the bottom to pass all the remaining material to another cascade, turning it over during the transfer. [Stat. ref.]

B. M. V.

**Mixing devices.** W. M. ROLPH (B.P. 398,324, 26.7.32).—A hand-operated device comprises a plunger which rotates a stirrer by means of an archimedean screw. Forms of vanes are described.

B. M. V.

**Determination of the percentage of water in wood, textiles, and other substances.** P. ROTHER and G. GRAU (B.P. 398,139, 26.5.33. Ger., 13.6. and 6.10.32).—The moisture bound by adsorption bears a definite relation to the humidity of the air in the interstices of the material; this air is therefore removed by suction and passed in contact with a hygrometric substance, *e.g.*, Co compounds that alter in colour.

B. M. V.

**Elutriating means.** M. A. LISSMAN, Assr. to INTERNATIONAL PRECIPITATION CO. (U.S.P. 1,894,623, 17.1.33. Appl., 7.3.29).—A pneumatic apparatus for, *e.g.*, measuring the amount of flour in cement comprises an elutriating chamber having an upper outlet connected to a filter in which the flour is collected, and a lower outlet connected to a small cyclone chamber in which the oversize grains are scrubbed by the entering air.

B. M. V.

**Filtering.** J. and L. R. DE LA TORRE (J. DE LA TORRE E HIJO (B.P. 398,055, 18.11.32).—A filter medium for non-greasy liquids comprises textile material every individual fibre of which has been thoroughly degreased.

B. M. V.

**Filtering or straining apparatus.** AUTO-KLEAN STRAINERS, LTD., and W. R. BELDAM (B.P. 397,907, 2.3., 15.9., and 25.11.32).—A filter having an internal moving-vane pump producing local back-flow is described.

B. M. V.

**[Stream-line] filters.** STREAM-LINE FILTER CO., LTD., H. S. HELE-SHAW, and S. BAKER (B.P. 397,867, 25.2. and 11.3.32).—A filter pack composed of sheets or strips of celluloid, synthetic resin, metal, or other impervious material has the surface of the material made slightly matt. If strips are used they are wound into the form of a disc.

B. M. V.

**Manufacture of diaphragms, filters, and other porous bodies.** J. NEUHAUS (B.P. 398,349, 6.10.32).—A mixture of fibres (asbestos, hemp, wood, etc.), rubber, and S is made by known means and partly vulcanised, then disintegrated into flocks, placed loosely in moulds, and revulcanised under slight or zero pressure. The proportion of fibre may range from 10%, giving strong filter plates of moderate porosity, to 33.5%, giving high porosity and low strength.

B. M. V.

**Perforate body adapted for use in aerating or carbonating liquids, for separating liquids from solids, for emulsifying immiscible ingredients, and other like purposes.** C. F. MORGAN, and WATNEY, COMBE, REID & Co., LTD. (B.P. 398,114, 17.3.33).—An element for subdividing gas comprises a no. of superposed plates having notched annular ribs on one face of each plate.

B. M. V.

**Evaporators.** J. STRACHAN, J. HOLMES, and H. A. KINGCOME (B.P. 398,004, 2.7.32).—The steam-heated tubes of a barometric-type evaporator are attached to the vapour drum by inclined flanges, and are supported near their lower ends by spring suspensions to permit free expansion and ready removal.

B. M. V.

**Means for supplying air or gas in a more or less finely-divided state to liquids.** ACTIVATED SLUDGE, LTD., E. R. JONES, A. B. DENTON, and J. A. COOMBS (B.P. 397,841, 22.2.32).—A method of fastening porous stones, or other elements for subdividing gas, in a metallic tray forming a blast chamber is described.

B. M. V.

(A) **Washing of gas.** (B, C) **Apparatus for washing gas or vapours.** R. R. HARMON, and PEABODY ENG. CORP. (B.P. 397,862 and 397,869—70, 22.1.32).—(A) The gas is stripped of the larger particles of dust in a no. of first stages comprising hot-H<sub>2</sub>O sprays above the dew point and baffles, steam is added if desired, and the H<sub>2</sub>O is re-used after settling. In a no. of second stages dew is formed by condensation and the remaining dust (forming nuclei of the dew drops) is removed by other barriers of H<sub>2</sub>O. (B) The centrifugal spraying and spray-collecting means are further described, and, in (C), partitions with baffled orifices.

B. M. V.

**Recovery of gases or vapours by solid adsorbent substances with recovery of heat units.** SOC. DE RECHERCHES ET D'EXPLOIT. PÉTROLIFÈRES (B.P. 398,010, 26.7.32. Ger., 1.8.31).—The apparatus comprises an adsorption mass (A) and a heat-accumulating mass (B) in the same casing. The gas to be scrubbed is passed through B before A; to strip A steam is passed through A and then B, and to dry A air or gas is passed through B and then A.

B. M. V.

**Removal of acid constituents from gases and rendering said constituents utilisable.** METALLGES. A.-G. (B.P. 396,969, 9.11.32. Ger., 10.11.31).—Flue gases containing small amounts of SO<sub>2</sub>, HCl, HF, or SiF<sub>4</sub> are washed with aq. suspensions of finely-divided coal ash or flue dust, whereby the basic constituents of the latter are converted into sulphates, chlorides, or fluorides which can subsequently be recovered.

A. R. P.



**Apparatus for heating.** J. A. ROULTON, ASSR. to ATLANTIC REFINING CO. (U.S.P. 1,906,422, 2.5.33. Appl., 14.11.31).

**Separating particles from fluids. Detecting etc. dusts suspended in gases.**—See XI.

## II.—FUEL; GAS; TAR; MINERAL OILS.

**New problems of the coal industry. Interest of modern methods for the utilisation of coal to the Czechoslovak Republic.** C. BERTHELOT (Chim. et Ind., 1933, 29, Spec. No., 350—361).—A review of the present position. E. S.

**Modern methods of cleaning and drying coals.** C. BERTHELOT (Chim. et Ind., 1933, 29, Spec. No., 362—370).—A comparison of modern methods for the cleaning of coals and drying of the fines. E. S.

**Improvement of lignite by heating in presence of steam under pressure.** A. RČIČKA and B. G. ŠIMEK (Chim. et Ind., 1933, 29, Spec. No., 381—412).—The physico-chemical transformations occurring in lignite (I) on drying in steam under pressure and the most favourable conditions for such drying are discussed. A new procedure is described in which the (I) is heated in the presence of  $H_2O$  by admission of superheated steam at pressures of 20—35 kg./sq. cm., and a comparison is made with other similar processes. Results are given to show the influence of different factors on the choice of the conditions of drying, and the effect of the latter on the dried product. A semi-industrial installation, consisting of an autoclave of 1 cu. m. capacity, and an industrial installation with a capacity of 400 tons per day are described. The advantages accruing from the use of dried (I) are discussed. E. S.

**Electrochemical extraction of humic acids.** B. G. ŠIMEK and J. LUDMILA (Chim. et Ind., 1933, 29, Spec. No., 371—380).—A method for the extraction and determination of the humic acids (I) in coals, soils, etc. is described. The apparatus consists of an electro-osmotic cell in which the space between the anode (A) and cathode (C) diaphragms is divided by a suitable filter. The powdered material is digested with aq.  $NH_3$  and the whole then introduced into the compartment surrounding C. On passing a suitable current the extracted (I) pass to A whilst the extraneous material is retained on the filter. All operations are carried out in an atm. of  $N_2$ . The (I) liberated on acidification are dried in vac. at  $105^\circ$ . Practical applications are cited and results given for 8 lignites and 2 soils. Caking power in the lignites is characterised by a high (I) content. E. S.

**Analysis of coking coals.** F. COUFALÍK (Chim. et Ind., 1933, 29, Spec. No., 327—336).—The coal (1.5 kg.) is carbonised in a vertical, electrically heated retort. Tar is removed from the gas by an electrostatic precipitator,  $NH_3$ ,  $H_2S$ , and  $C_{10}H_8$  are eliminated by washing with suitable reagents, and the  $C_6H_6$  is adsorbed by active C. Results are reproducible and show good agreement with those obtained in large-scale practice. E. S.

**Recent developments in coking practice.** H. KOPPERS (J. Inst. Fuel, 1933, 7, 13—28).

**Recent developments in the coking process.** K. BUNTE (Gas- u. Wasserfach, 1933, 76, 685—691).—Foxwell's method of studying the plastic range of a coking coal (B., 1924, 737, 738) has been applied to a no. of German coals. The influence of storing and of blending coals on their plastic properties, and the relation between the coking properties of a coal, its bitumen content ( $\alpha$ ,  $\beta$ , and  $\gamma$  constituents), and the rate of gas evolution during carbonisation, are discussed (cf. B., 1932, 294; 1933, 771). A. B. M.

**Evaluating the coking capacity of coals.** K. DREES and G. KOWALSKI (Gas- u. Wasserfach, 1933, 76, 653—660).—A standard coking test using a closed metal crucible is described. The results obtained depend on the rate of heating, and as the process of carbonisation is in practice slow it is suggested that comparable results can be obtained only if experiments are carried out after heating to  $300^\circ$ ,  $350^\circ$ , and  $375^\circ$  and at  $350^\circ$  for 1, 2, and 3 hr. Investigation of Francis and Wheeler's method (Fuel, 1931, 10, 171) shows that the  $KMnO_4$  no. for different coals becomes similar after heating for  $> 1$  hr. at  $350^\circ$  although the untreated coals gave very different vals. This no. increases again in coals heated to  $> 375^\circ$ , reaches a max. at  $425$ — $450^\circ$ , and then decreases. The max. val. is associated with the evolution of tarry matters. The gas-evolution method of Foxwell (J.S.C.I., 1921, 40, 193 r) has been applied to the same coals and the % volatile matter evolved in the range  $350$ — $500^\circ$  determined. The above methods and the  $C_5H_5N$  extraction method are compared. R. N. B.

**Coke producer with withdrawal of the fused slag.** A. HECZKO (Chim. et Ind., 1933, 29, Spec. No., 420—426).—A slagging ash producer consuming 100—200 tons of coke per day is described, which has been in operation since 1926. Cost of installation and operation together with thermal balance are given. E. S.

**Dry distillation of residue of waste sulphite liquor.** M. PHILLIPS (Ind. Eng. Chem., 1933, 25, 991—993).—When distilled up to  $400^\circ$  in  $CO_2$  the aq. fraction (22.3%) contained  $AcOH$ ,  $COMe_2$ , and  $MeOH$ , and the oily fraction (1.5%)  $AcOH$ , guaiacol, and 4-propyl-guaiacol, together with S compounds. The carbonised residue and gas were 55 and 21.2%, respectively. The yield of oil was small as compared with that from the distillation of lignin. C. A. K.

**Reactivity of coke.** R. KASSLER (Chim. et Ind., 1933, 29, Spec. No., 315—326).—The reactivity (R) to  $CO_2$  is determined by measuring the vol. increase produced on passing a known vol. of gas several times over the coke at  $1000^\circ$ . Four cokes, charcoal, and graphite were examined. Increase in the rate of gas flow and decrease in the particle size produce only a slight increase in R as thus measured. Extraction of ash with  $HCl$  and  $HF$  decreases R. A relation exists between the R to  $CO_2$  at  $1000^\circ$  and the power of decomposing  $H_2O_2$  at  $20^\circ$ . E. S.

**Mechanical analysis of coke.** M. HEPNER (Chim. et Ind., 1933, 29, Spec. No., 337—343).—Factors affecting the physical properties of coke are discussed and the various methods for determining these properties reviewed. E. S.



**Dependence of the properties of coke on the conditions of its production.** I. W. J. MÜLLER and E. JANDL (Brennstoff-Chem., 1933, 14, 341—347).—"Model" cokes have been prepared by the carbonisation of a selected coal blend in a laboratory coking apparatus, at temp. from 700° to 1300°, using either a slow (10°/min.) or a rapid rate of heating. The reactivities of the cokes were determined by the authors' method (B., 1931, 660). The reactivity ( $R$ ) of the slowly carbonised cokes increased slightly with time of carbonisation, passed through a max., and then decreased; the total variation was slight (over 16 hr.) with the 700° coke, but the decrease with time became more marked as the carbonisation temp. ( $T$ ) rose;  $R$  also decreased with rising  $T$ . With the rapidly carbonised cokes (I) the fall in  $R$  with rising  $T$ , and the fall in  $R$  of the high-temp. cokes with time, were still more marked. The results when represented on a  $R$ - $T$ -time space model gave a smooth continuous surface. The  $R$  curves of two industrial cokes resembled those obtained with (I). A. B. M.

**Improvement of coke structure by petrographical treatment of the coal.** M. SPETL (Chim. et Ind., 1933, 29, Spec. No., 344—349).—Washed dust from the coal, consisting chiefly of clarain ( $C$ ), is remixed with a portion of the washed coal; this blend on carbonisation gives a hard, dense, coke. The other portion of the coal thus partly freed from  $C$  gives a more friable and bulky coke. E. S.

**Preparation of gas-adsorbent carbon by water-vapour activation.** E. DEISENHAMMER (Chem.-Ztg., 1933, 57, 781—782).—A description of existing practice. E. S. H.

**Manufacture and testing of gas-absorbing charcoal, with special reference to the zinc chloride method.** Y. H. LI, T. S. MA, J. Y. JEN, and S. H. LEI (J. Chinese Chem. Soc., 1933, 1, 64—78).—The raw material [walnut ( $A$ ) or coconut ( $B$ ) shell, bass or elm wood] was boiled with an activating solution [ $ZnCl_2$  in aq.  $HCl$  or  $H_2SO_4$ , in one case with added sugar and  $Ni(NO_3)_2$ ;  $FeCl_3$  or  $AlCl_3$  in aq.  $HCl$ ; dil.  $H_2SO_4$  or  $H_3PO_4$ ; or aq.  $Na_2CO_3$ ,  $NaOH$ , or  $NaHSO_3$ ], carbonised, activated by heating in steam (with or without air), boiled with dil.  $HCl$  to remove  $ZnO$  etc., and dried by heat. The adsorbent power of the product for  $CCl_3 \cdot NO_2$  vapour was determined.  $ZnCl_2$  was found to be the only satisfactory activating agent.  $B$  gave the most active  $C$ , but required longer boiling with  $ZnCl_2$  than  $A$ . The woods were distinctly inferior. D. R. D.

**Decolorising power of activated charcoal.** Y. H. LI and T. S. MA (J. Chinese Chem. Soc., 1933, 1, 79—91).—The gas adsorption (I) and decolorising power (II) of animal charcoal ( $A$ ) and various types of activated charcoal ( $B$ ), prepared as described in the preceding abstract, have been compared. Dark glutamic acid hydrochloride solution (prepared from wheat gluten and  $HCl$ ) is a better solution than methylene-blue for testing (II). There is no close correlation between (I) and (II).  $B$  has greater (II) than  $A$  when used in large quantities, but the reverse is true when the proportion of charcoal taken is small. For decolorising very dark solutions it is most economical to decolorise partly by means of  $A$  and finish the work with  $B$ . D. R. D.

**Hydrogenation of coal and tar.** J. G. KING (Chim. et Ind., 1933, 29, Spec. No., 592—598).—A résumé. E. S.

**Analysis of fuels and mine gases by means of the microdynamograph.** J. BUCHTALA (Chim. et Ind., 1933, 29, Spec. No., 599—600).—Mixtures of air with a gas or vapour are exploded in a cylinder closed by a piston the movement of which is traced graphically. Examples are given of the application to the analysis of EtOH-benzene mixtures and determination of  $CH_4$  in mine gases. The apparatus is easily portable. E. S.

**Distribution of sulphur in [gas] purification.** W. ZWIEG and F. KOSSENDEY (Gas- u. Wasserfach, 1933, 76, 644—648).—The %  $H_2O$  and S in an 80 : 20 mixture of Luxmasse and bog ore used for purifying coal gas has been determined at 25 equally spaced positions in the cross-section of each of the layers of a 4-layer box-purifier working under standard conditions. The highest  $H_2O$  and lowest S content occurs in the corners of the top layer farthest from the gas outlet. This is attributed to temp. differences, and could probably be avoided by using circular-shaped purifiers. R. N. B.

**Profitable by-product sulphur from city gas.** R. S. McBRIDE (Chem. Met. Eng., 1933, 40, 398—401).—About 99% of the  $H_2S$  is removed from coal gas by scrubbing with aq.  $NH_4$  thioarsenate containing 0.5 wt.-%  $As_2O_3$ . Air is then blown through the solution and the slurry of S formed is skimmed off and collected on a rotary filter. The filter-cake is melted in a steam-jacketed autoclave, and the molten S is separated and cast into blocks; these contain < 0.5%  $As_2O_3$  and are used for making  $H_2SO_4$ . Gaseous  $NH_3$  or dil. aq.  $NH_3$  liquor is added to the filtrate, which is then re-used. The economics and operating data of the process are given. D. K. M.

**Methane indicator for direct detection of fire-damp in mines.** G. DUNKEL (Chim. et Ind., 1933, 29, Spec. No., 413—419).—Easily portable and in the form of a pistol, the apparatus consists of sampling, reaction, and absorbing chambers, and a manometer ( $M$ ). The  $CH_4$  is oxidised by passing the sample over a Pd spiral heated electrically (external accumulator). The  $H_2O$  formed is absorbed by  $CaCl_2$ , and the decrease in pressure, read on  $M$ , gives the  $CH_4$  concn. With concns. < 6% the method is quant., but for greater concns. qual. only. Determinations occupy 65—70 sec. and the results are not affected by small variations in temp. and pressure. In operation it is as safe as the miner's electric lamp. E. S.

**Neunkirchen [gasholder] explosion.** K. BUNTE, H. BRÜCKNER, and W. LUDEWIG (Chem. Fabr., 1933, 6, 383—388).—A description is given of the works and gasholder involved. The explosion and fire adjacent to the holder probably caused part of the gas to be forced above the piston, and the resulting explosive mixture was ignited by the walls, which had certainly reached a temp. > 420°; it is most improbable that explosion occurred beneath the piston. The merits of the  $H_2O$ -less gasholder are discussed. Tables are given of the explosion limits and velocity of propagation of flame in many binary gas-air mixtures. H. F. G.



**Chemistry of some menilite shales from the borders of the Flysch in East Carpathia.** E. CASIMIR [with M. DIMITRIU and V. PAȘCA] (Petroleum, 1933, 29, No. 37, 1—4).—Seven samples of shale have been examined. The content of org. material was high (up to 14%) in the shales rich in clay, but negligible in those rich in  $\text{CaCO}_3$ . On carbonisation at  $650^\circ$  the org. material (C 90, H 7.25%) yielded: oil 27, gas 15, coke 58%. The shales are probably similar in origin and mode of formation to the sapropelite deposits in salt lakes.

A. B. M.

**Physical and chemical properties of North German petroleum.** G. KEPPELER (Petroleum, 1933, 29, No. 37, 6—7).—Data relating to 8 petroleum are tabulated:  $d^{20}$  ranged from 0.9518 to 0.8475, the "olefinoid" content (*i.e.*, constituents extracted by 92%  $\text{H}_2\text{SO}_4$ ) from 59 to 12%, the "aromatoid" content (constituents extracted by 100%  $\text{H}_2\text{SO}_4$ ) from 12 to 53%, and the paraffin-naphthene content from 25 to 35%. The petroleum of lowest  $d$  contained 23% of constituents boiling to  $150^\circ$  and 12% boiling from  $150^\circ$  to  $200^\circ$ .

A. B. M.

**Recent developments in motor fuels and in engines for motor transport.** WA. OSTWALD (Brennstoff-Chem., 1933, 14, 349—353).—The production of anti-knock fuels for petrol engines and of Diesel fuel oils of low ignition temp., the increase of efficiency of combustion in the cylinder by improved design of the cylinder head, and recent developments in the design of the 2-stroke engine, are discussed.

A. B. M.

**Effect of metallic (lead) dope on carbonisation of oil in the combustion space of an engine.** R. O. KING (Engineering, 1933, 136, 183—186).—The accumulation of low-temp. oxidation products in mineral oil used for lubricating main bearings and big ends in internal-combustion engines may cause increased viscosity ( $V$ ) and fluid friction ( $F$ ), or, if  $V$  is not entirely the determining factor, a reduction of  $F$ . These two opposite effects were seen in journal-bearing experiments when the lubricating val. diminished at  $40^\circ$ , remained const. at  $100^\circ$ , and markedly improved at  $150^\circ$ . In order to control oxidation, oil is doped with  $\text{PbEt}_4$  and  $\text{C}_2\text{H}_4\text{Br}_2$ . It is shown that (1) the effect of the dope persists for the usual life of the oil; (2) doped oil (I) used in journal-bearing experiments lasting 96 hr. is free from decomp. products, whilst undoped oil (II) is heavily impregnated; (3) 12 c.c. of dope per gal. are as effective as 45 c.c.; (4) 1% and 2% of dope prevents oxidation of oil vapour at temp. near the flash point of the oil, whilst (II) is oxidised to hard carbonaceous products; (5) the performance of a motor-cycle engine run at 30—35 m.p.h. for 500 miles on (I) is as good at the end as at the beginning, only a thin film of soft C forming on the cylinder head, the condition of the piston rings being unusually good and the oil free from sludge; (6) there is a 20% reduction in oil consumption with (I); (7) in bench tests of a 4-stroke engine with (II), lubricating failure occurs in at least two stages, each being marked by recognisable changes in engine speed, power, and temp. Running is unstable and the first permanent lubrication failure occurs in 2 hr. A higher output is reached and maintained

for 10 hr. with (I) before failure occurs. In both trials, the first lubricating failure is followed immediately by a partial recovery succeeded by a second failure, after which the engine runs for an indefinite period at reduced power. The piston ring condition corresponds with the engine performance. The Pb content of the oil was determined by X-ray shadowgraphs, and the presence of deposits by photographs by infra-red radiation.

V. P. P.

**Course of combustion of hydrocarbon vapour-air mixtures.** H. SCHILDWÄCHTER (Angew. Chem., 1933, 46, 587—592).—A bomb is described which is so fitted that records are obtained on the same film of time, sparking time, pressure, time that flame reaches each of two points, and sound. Using  $\text{C}_6\text{H}_{14}$ -air mixtures initially at 10 atm. and  $150^\circ$ , the combustion and explosion velocities were max. when the air excess no. ( $\lambda$ ) was 0.9. The max. pressure increased slowly with decreasing  $\lambda$  to  $\lambda = 0.9$ , and below this it increased rapidly. The sound started shortly before the flame reached the bottom of the bomb. The max. pressure was 4—15% below the calc., the difference increasing as  $\lambda$  decreased. The max. temp. was calc. from the pressure.

A. G.

**Composition of Ural petroleum (Perm).** N. D. ZELINSKY and J. K. JURIEV (Brennstoff-Chem., 1933, 14, 347—349; cf. B., 1931, 661).—The fraction of b.p.  $150$ — $240^\circ$  was treated with Hg salts to remove S compounds, and fractionated over "mol." Cu. The composition of each fraction was determined by (a) removing the aromatic hydrocarbons (I) by means of  $\text{H}_2\text{SO}_4$ , (b) subsequently dehydrogenating the hexahydro-aromatic hydrocarbons (II) over Pt at  $310^\circ$  and removing the aromatics formed, and (c) determining the  $\text{NH}_2\text{Ph}$  point of the residual oil. The vals. for the wt.-% of (I), (II), other naphthenes, and paraffins varied from 72.2, 8.7, 6.5, and 12.6 ( $150$ — $170^\circ$ ), respectively, to 47.9, 7.8, 17.9, and 26.4 ( $225$ — $240^\circ$ ), respectively. The high (I) content of this oil is noteworthy. Mono-substituted  $\text{C}_6\text{H}_5$  derivatives are present even in the highest-boiling fraction; the di-substituted derivatives present consisted principally of *m*- and *p*-compounds.  $\text{C}_{10}\text{H}_8$  and its hydrogenated derivatives were absent.

A. B. M.

**Pressure-temperature and low-pressure total heat relationships of petroleum fractions.** E. G. RAGATZ, E. R. MCCARTNEY, and R. E. HAYLETT (Ind. Eng. Chem., 1933, 25, 975—979).—A method of identifying the equiv. pure cut of a wide-cut fraction is described. The crit. point for such a cut constitutes the common point of convergence of the dew lines of all similar wide-cut fractions of equal sp. gr. A means of calculating the total heats of low-pressure petroleum fraction vapours is developed.

E. S. H.

**Action of acetylene in petroleum cracking gas on copper and brass.** J. S. SALKIND and B. A. VOVSİ (J. Appl. Chem. Russ., 1933, 6, 677—680).—Cracking gas, containing 1.68%  $\text{C}_2\text{H}_2$ , attacks Cu and brass to a negligible extent at room temp., and not at all at  $80^\circ$ .

R. T.

**Hydrogenation of petroleum.** H. S. GARLICK (Ind. Chem., 1933, 9, 226—228, 278—280).—The



hydrogenation of petroleum is indicated for the improvement of colour and quality, gum stabilisation, elimination of S, production of special products, and conversion of residues into oils. Details of manufacturing operations are given.

D. K. M.

**Pyrolysis of the lower paraffins. II. Production of olefines in baffled quartz tubes.** A. CAMBRON and C. H. BAYLEY (Canad. J. Res., 1933, 9, 175—196; cf. B., 1933, 338).—Calculation of approx. crit. velocities for the lower paraffins shows that under the usual conditions of pyrolysis the flow is streamline, whereas with turbulent flow the heat exchange between the gas and the tube walls should be more efficient, since the surface film of gas breaks down. Turbulent flow is produced by baffles (*A*), and the effect of the no. and diam. of the *A* on the turbulence is discussed. The degree of turbulence is approx. independent of the no. of *A* within certain limits. Experiments with paraffins from  $C_2$  to  $C_6$  show that with baffled tubes a greater yield of olefine, greater freedom from side reactions, and a greater thermal efficiency than in unbaffled tubes under similar conditions are obtained. The decrease in thermal efficiency with increasing mol. wt., and the decrease in the velocity coeff. with rise in temp. are discussed.

A. A. L.

**Distillate obtained in shell stills on distillation of paraffinic heavy bottoms to coke.** GROZNI PETROL. RES. INST. (Grozni Neft., 1932, 2, No. 11—12, 66—67).—Yields and characteristics are recorded.

CH. ABS.

**Rate of dissolution of methane in quiescent liquid hydrocarbons.** R. D. POMEROY, W. N. LACEY, N. F. SCUDDER, and F. P. STAPP (Ind. Eng. Chem., 1933, 25, 1014—1019).—Apparatus for determining the diffusion consts. and solubilities of gases in liquids at pressures up to 30 atm. is described. Results are given for  $CH_4$  in isopentane. The abs. diffusion const. may be determined with an error  $\pm 4\%$ ; the val. is not affected by concn. of  $CH_4$  at pressures  $< 30$  atm.

E. S. H.

**Comparison of high-solvent value petroleum solvents.** CLEVELAND CLUB (Amer. Paint & Varnish Manufs.' Assoc., Circ. 423, Nov., 1932, 4 pp.).—A range of proprietary petroleum solvents (produced by selective extraction or by hydrogenation of "cracked" unsaturated hydrocarbons) were examined. The following properties are tabulated: distillation range, evaporation rate,  $Me_2SO_4$  val., kauri-BuOH val.,  $NH_2Ph$  point, and viscosities of various synthetic resin-linseed and -tung oil varnishes made with these solvents.

S. S. W.

**Oil emulsions and their decomposition by means of the electric current.** N. A. RYAZANOVSKI (Grozni Neft., 1932, 2, No. 11—12, 45—52).—Grozni oil emulsions are of the hydrophobic type. An electrical de-emulsifier is described.

CH. ABS.

**Analysis of hydrocarbon oils.** R. H. GRIFFITH and H. HOLLINGS (J. Inst. Petroleum Tech., 1933, 19, 701—712).—The  $NH_2Ph$  point (I)-b.p. curves for the residue of paraffins obtained by treating the gas oil fractions of several petroleum oils with oleum and for the naphthenes obtained by extracting a mixture of paraffins and naphthenes isolated from a Borneo gas oil

with  $COMe_2$  and  $SO_2$  are linear. For mixtures of naphthenes and paraffins the observed (I) agrees with that calc. from the composition of the mixture. The addition of each 1% of naphthene to paraffins lowers the (I) by  $0.4^\circ$ . The mean b.p. of a naphthene-paraffin mixture may be taken as that of oil from which the mixture was separated. The (I)-b.p. curve is linear for natural oils and oil obtained by the hydrogenation of low-temp. tar, but is curved for cracked oils.

D. K. M.

**Mechanism of the alteration of mineral oils.** T. SALOMON (Chim. et Ind., 1933, 29, Spec. No., 562—591).—Oil at  $115^\circ$ , with an immersed Cu spiral, is exposed to the air. The time required for the first production of turbidity and the rate at which a deposit is produced are noted. The total amount of deposit varies with the time, first parabolically, then linearly. A complete curve may consist of several such curves, superimposed to various extents, and the meaning of these is discussed. The deposit is less for light fractions than for heavy, similarly refined, from the same oil. The effect of washing with  $H_2SO_4$  depends not only on the total amount of acid used, but also on the no. of separate washes, a large no. of small washes giving the more stable product. Rapid removal of acid sludge, complete neutralisation of the acid-washed oil, and thorough washing of the neutralised oil increase the stability of the refined product. Oils in internal-combustion engines apparently alter much more rapidly in use than those in transformers and steam turbines.

T. H. B.

**Specific heat of [viscous] oils.** LOUIS and ROLLIN (Chim. et Ind., 1933, 29, Spec. No., 559—561).—The sp. heats of Alsatian mineral oils of various viscosities, measured by an electrical-input calorimeter, ranged from  $0.45$  to  $0.50$  at  $30^\circ$ , increased linearly with temp. over the range  $30$ — $90^\circ$ , and were highest for the lighter oils from the same crude.

T. H. B.

**Emulsified paraffin wax sizes.** R. M. COBB, D. S. CHAMBERLIN, and B. A. DOMBROW (Paper Trade J., 1933, 97; T.A.P.P.I. Sect., 111—114).—By the use of a virtually non-aq. emulsifying base (not disclosed) it is possible to prepare stable paraffin wax emulsions without the need for high-speed emulsifying equipment. A ratio of 4 pts. of wax (m.p.  $50^\circ$ ) to 1 pt. of base is recommended. The emulsion is stable indefinitely, even in hard  $H_2O$ . The average droplet diam. is  $0.5 \mu$ ; this degree of dispersion approaches that of rosin size ( $0.1 \mu$ ). Wax and wax-rosin dispersions made by other methods are much coarser. The claim that wax sizes are more efficient than is rosin size at low temp. is not confirmed, nor is it found that they are less susceptible to  $pH$  variations.

H. A. H.

**Production of highly insulating oils.** TYPEKE (Erdöl u. Teer, 1933, 9, 29; Chem. Zentr., 1933, i, 2202).—Addition of (MeOH) extracts of axle oil distillates to refined white oil reduces oxidisability without causing the formation of sludge.

A. A. E.

**Vacuum distillation applied to [the testing of] lubricating oil.** J. TILMAN (Chim. et Ind., 1933, 30, 518—524).—A 2-stage oil pump allowed pressures as low as  $0.2$  mm. Hg to be obtained even with varnished



rubber connexions. The vac. was measured both directly and with a Geissler tube, and the sample was heated electrically. Several arrangements of the apparatus, which occupies a space of only  $50 \times 50$  cm., are described. Typical distillation curves over the range  $100-300^\circ$  at pressures of  $0.5-1.0$  mm. are given. No discontinuities were noted, but it is considered that an oil can be characterised by the mean slope of the distillation curve between 2 arbitrary points rather than by such qualities as  $d$ ,  $\eta$ , and flash point. No decomp. occurred during distillation under these conditions.

C. I.

**Testing of machine lubricating oil.** ANON. (Chem.-Ztg., 1933, 57, 782—784).—Apparatus is described in which the effects of temp., pressure, and velocity on the lubricating conditions can be studied separately. Results are given for typical oils.

E. S. H.

**Ageing of oil in transformers.** TYPKE (Erdöl u. Teer, 1933, 9, 61; Chem. Zentr., 1933, i, 2202).—The use of phenyl- $\alpha$ -naphthylamine as protective agent for Russian transformer oil was unsuccessful.

A. A. E.

**Chemical treatment of rotary drilling fluids. Stabilisation of weighted dispersions: effect of  $p_H$  value.** H. A. AMBROSE and A. G. LOOMIS (Ind. Eng. Chem., 1933, 25, 1019—1022; cf. B., 1932, 969).—The weighted fluids are discussed from the colloid viewpoint. The curve relating the amount of the commercial product "Baroid" remaining in suspension in a 25% dispersion in  $H_2O$  to the  $p_H$  exhibits two maxima, one on the acid side and the other on the alkaline. The stability of the suspension is greatest at  $p_H$   $10.5-11.5$ . This region of  $p_H$  is advisable for drilling practice; moreover, the slight degree of alkalinity serves to reduce corrosion of the drill.

E. S. H.

**Hydrogenation.**—See I. Removing  $C_5H_5S$  from  $C_6H_6$ .—See III. Determining  $O_2$  in gases.—See VII. Bituminous road-making emulsions. Timber preservatives.—See IX. Oilfield  $H_2O$  analysis. Trade wastes from gasworks.—See XXIII.

See also A., Oct., 1016, Spontaneous ignition of  $C_4H_{10}$ -air mixtures. 1032, Oxidation of paraffins to fatty acids.

## PATENTS.

**Briquetting of coal.** C. NIMZ (H. DAW & Co.) (B.P. 398,007, 7.7.32. Ger., 22.1.32).—The coal dust is mixed with  $1.5-2\%$  of sorghum meal and the mixture is treated with steam at  $120-150^\circ$  and briquetted.

A. B. M.

**Apparatus for low-temperature carbonisation.** T. H. PARRY, W. JOHNSON, and H. A. S. GOTHARD (B.P. 398,160, 29.1.32).—Coal slack is mixed with tar or oil and is carried into a horizontal retort ( $R$ ) by means of a drag-link conveyor ( $C$ ) which carries sealing devices for closing the ends of  $R$  when the charged  $C$  has been drawn completely thereinto. The links of  $C$  form a series of cells which are charged with the mixture from a feed chute as  $C$  moves across a feed-table on its way to  $R$ . When carbonisation is completed  $C$  is withdrawn and the coke is discharged through an opening into a cooling hopper, coke adhering to the  $C$  links being removed by means of a pivoted wt. which can be given a hammering action, or by similar means.

A. B. M.

**Distillation retort.** G. ZOROS (U.S.P. 1,897,877, 14.2.33. Appl., 14.3.29. Fr., 15.9.28).—Solid fuel, e.g., lignite, is fed continuously downwards through a shaft fitted with staggered, downwardly-inclined baffles. The centre of the shaft is occupied by a vertical combustion chamber ( $C$ ) surrounded by a bell ( $B$ ). Fuel dust is subjected to incomplete combustion in the central chamber and the gases produced are passed through the charge; part of the gases are then passed, by means of a blower, down the annular space between  $C$  and  $B$  and thence again through the charge. The carbonised fuel is cooled by means of a  $H_2O$ -spray before being discharged from the bottom of the shaft. The heating val. of the fuel is enhanced by the process, which by suitable regulation yields only small quantities of gas and tar.

A. B. M.

**Destructive distillation and carbonisation of waste matter.** O. W. WAHLSTROM (U.S.P. 1,898,326, 21.2.33. Appl., 16.4.32).—The material is compressed into bales and bound. The bales are loaded into airtight metal containers which are then evacuated. The charged container ( $C$ ) is introduced into a retort ( $R$ ) and connected through a valve with a separator, condensing system, etc. The valve is opened when the pressures on both sides thereof are equalised. When carbonisation is completed  $C$  is withdrawn from  $R$ , allowed to cool, and the charcoal therein is discharged and subsequently refined.

A. B. M.

**Coking process.** G. P. BARTHOLOMEW, ASSR. to AMER. SMELTING & REFINING CO. (U.S.P. 1,894,438, 17.1.33. Appl., 20.10.28).—Coal tar is sprayed into a chamber ( $A$ ) heated to  $700-750^\circ$  by the previous combustion of coke therein, whereby the volatile constituents (creosote) are removed as vapour while the residue is partly coked. When the temp. has fallen to approx.  $400^\circ$  air is introduced into  $A$ , whereby a part of the residue is burned, the heat developed being utilised in completing the coking of the remainder and in again raising the temp. of  $A$  to  $700-900^\circ$ . After removal of the coke the cycle of processes is repeated. A convenient form of  $A$  is one resembling a bee-hive coke oven.

A. B. M.

**Oil-gas generator.** E. E. BUSSEY, ASSR. to BUSSEY FUEL GASIFIER CORP. (U.S.P. 1,896,220, 7.2.33. Appl., 12.10.26. Renewed 18.6.32).—Oil vapours are cracked to gas by being passed through a jacketed chamber ( $A$ ) packed with metallic wool and heated by the passage of hot gases, e.g., exhaust gases, through the jacket and through heating tubes traversing  $A$ . The vapours may be preheated by means of an auxiliary electrical heater. The device permits the use of less readily volatile fuels in internal-combustion engines.

A. B. M.

**Apparatus for manufacture of combustible gas.** H. O. LOEBELL, ASSR. to H. L. DOHERTY (U.S.P. 1,896,799, 7.2.33. Appl., 12.10.26. Cf. U.S.P. 1,826,007 and 1,837,226; B., 1932, 791, 874).—The fuel is passed continuously down through a vertical shaft ( $S$ ) and a high-temp. zone is maintained in the mid-portion of the column by continuously blasting through it a mixture of preheated air and steam. The blast inlet and blast-gas offtake are arranged at the circumference of  $S$  but at different heights thereof. A small proportion of the



blast gases is passed through the upper layer of fuel, thereby carbonising it, the enriched gas so produced leaving by a gas offtake mounted axially in the top of *S*. The coke is discharged continuously from the bottom of *S* by means of a rotating, eccentrically-shaped cone platform and is quenched by a spray of  $H_2O$  or steam. A. B. M.

**Removal of ammonia from fuel gas.** KOPPERS CO. OF DELAWARE, ASSEES. OF F. DENIG (B.P. 397,537, 19.2.32. U.S., 13.1.32).—The gas is scrubbed in known manner to remove one half of the  $NH_3$ , which is converted into  $NH_4HSO_4$  (I) by treatment with  $SO_2$  and  $O_2$  in the presence of  $H_2O$ . The residual  $NH_3$  in the gas is removed by scrubbing it with aq. (I), which is thereby converted into  $(NH_4)_2SO_4$ . A. B. M.

**Desulphurising of coal-distillation gases.** GEWERKSCHAFT M. STINNES (B.P. 398,047, 24.10.32. Ger., 23.10.31).—The gas, to which air and/or steam are/is added, is passed through at least 3 boxes charged with  $Fe_2O_3$ ; after it has left the first or second box its temp. is raised  $\leq 10^\circ$ , e.g., to  $30-60^\circ$ , and it is treated in the remaining boxes at this higher temp. The permissible max. rate of gas flow through the purifiers is thereby increased. A. B. M.

**Gas detectors.** BRIT. THOMSON-HOUSTON CO., LTD., ASSEES. OF A. L. MARSHALL (B.P. 397,815, 12.5.33. U.S., 12.5.32).—The presence of combustible gases, e.g.,  $CH_4$ , in air etc. is detected by passing the gas through a vessel within which is a porous-walled container (*A*). The gases diffuse through the walls of *A* and the combustible constituents are burned therein by means of an electrically-heated wire, means, e.g., a cooling coil, being provided within *A* for condensing the  $H_2O$  produced. The consequent diminution of pressure in *A* is automatically recorded. The outer vessel contains  $H_2O$  to keep the circulating gas moist. A. B. M.

**Production of acetylene.** R. W. MILLAR, ASSR. TO SHELL DEVELOPMENT CO. (U.S.P. 1,896,552, 7.2.33. Appl., 10.3.30).—Hydrocarbon gases, e.g., natural gas, are decomposed by means of an inverted flame, i.e., a flame produced by burning air or  $O_2$  in the gas, the ratio of hydrocarbon to  $O_2$  being 1.2–1.7. Liquid hydrocarbons may be similarly decomposed by means of an inverted flame burning below the surface thereof. A. B. M.

**Manufacture of acetylene from gases containing hydrocarbons.** J. Y. JOHNSON. FROM I. G. FARBENIND. A.-G. (B.P. 397,987, 30.5.32).—Gases containing hydrocarbons of the  $CH_4$  series, or other carbonaceous substances, are passed consecutively through two or more electric arcs, the energy supplied to each arc (per cu. m. of gas passed) being  $>$  that supplied to the preceding arc, so that the concn. of  $C_2H_2$  in the gases increases with each successive stage of the treatment. The  $C_2H_2$  formed is removed after each arc treatment, e.g., by conversion into  $MeCHO$ . The amount of C black formed is small. A. B. M.

**Production of gaseous mixtures and their application for use in reheating processes of steel and alloys.** R. HALKETT, H. A. FELLOWS, and J. BROWN (B.P. 397,640, 18.6. and 20.8.32).—Formation of scale

on steel and alloys during heat treatment is prevented by carrying out the process in an atm. formed by cooling combustion gases, removing the condensed  $H_2O$ , and then adding thereto some unburnt gas. A. B. M.

**Inert gas production.** A. D. HARRISON (U.S.P. 1,894,766, 17.1.33. Appl., 29.8.31).—An apparatus for producing  $CO_2-N_2$  mixtures from air and fuel is described, both constituents being under slight pressure before combustion and the proportions regulated by control of the booster fans. B. M. V.

**Distillation of tar.** S. P. MILLER, ASSR. TO BARRETT CO. (U.S.P. 1,898,980, 21.2.33. Appl., 2.11.28).—Tar is supplied under pressure to injector nozzles so designed that the jet aspirates hot coal-carbonisation gases from the collector main of a coke oven etc. into the injector chamber and thereby effects intimate mixture of the tar and gases. The pitch is withdrawn from the chambers, or may be recirculated for further distillation. The gases containing the oil vapours are passed to a condensing system. A. B. M.

**Conversion of coal-distillation products, particularly tar acids or products containing same, into valuable hydrocarbons substantially free from tar acids.** C. J. GREENSTREET (B.P. 397,468, 18.1. 8.3., and 1.9.32).—Creosote oils or coal-distillation products containing tar acids are mixed with  $\leq$  an equal wt. of mineral oil of approx. the same boiling range and the mixture is vaporised and passed over a catalyst at  $350-650^\circ$ . The catalyst, which should be in a form presenting an extensive surface to the vapour, may consist of Fe, Ni, Mo, Si, pumice,  $SiO_2$  gel, carborundum, etc. The initial mixture may be obtained by distilling a mixture of mineral oil and coal. A. B. M.

**Consolidation of tars and pitches.** O. REYNARD and E. H. THOMPSON (B.P. 397,484, 23.12.31).—Tar, tar oil, or pitch is heated to  $150-180^\circ$  with 4–10 wt.-% of conc.  $H_2SO_4$ , and compressed air is blown through the heated mixture. The heating may be effected under reduced pressure in an apparatus containing a system of shallow trays arranged in cascade form. The m.p. of pitch is thereby raised and the yield of pitch from tar is considerably increased. A. B. M.

**Oxidation of tars and like products.** SOC. POUR L'EXPLOIT. DES PROC. AB-DER-HALDEN (B.P. 398,112, 11.3.33. Fr., 17.3.33).—Preheated tar and air are passed continuously into a retort (*A*) containing a mass of the material undergoing oxidation, the preheating temp. being sufficiently high, e.g.,  $230-250^\circ$ , to enable the heat of reaction to maintain the mass at the required reaction temp., e.g.,  $280-300^\circ$ , without requiring any additional heat to be supplied to *A*. The oxidised product is removed continuously from *A*. Means are provided for cooling the issuing gases and vapours and returning any condensate to *A*. A. B. M.

**Purification and decolorisation of phenols and neutral oils.** G. T. MORGAN and A. E. J. PETTET (B.P. 397,847, 29.2.32).—Impure phenols or neutral tar oils from low-temp. or vertical-retort processes are heated, e.g., at  $100^\circ$ , with a small proportion, e.g., 1%, of  $CH_2O$  and with an alkali or acid catalyst, and then distilled. A. B. M.



**[Preparation of] mastic asphalts and agglomerates.** W. GRIFFITHS and F. FOULGER (B.P. 397,864, 22.2.32).—Soft pitch or similar bituminous binder is heated and mixed intimately with spent oxide or cyanide residue. The product may be used for the production of a road-making agglomerate (*A*) by mixing it with a suitable aggregate, *e.g.*, granite chippings. *A* may contain 6–7% of bituminous binder and 17–20% of spent oxide. A. B. M.

**Breaking of petroleum emulsions.** M. DE GROOTE, L. T. MONSON, and A. F. WIRTEL, Assrs. to TRETOLITE Co. (U.S.P. 1,897,574–5, 14.2.33. Appl., [A, B] 21.1.29).—(A) Petroleum emulsions of H<sub>2</sub>O-in-oil type are broken by introduction of sulphonic acids of aromatic hydrocarbons containing  $\leq 2$  alkyl, cycloalkyl, or aralkyl residues of  $< C_{12}$  each, the two together having  $> C_5$ , and giving a ppt. in  $\leq 1\%$  aq. solution with salts of the alkaline earths. Such compounds are prepared by sulphonating a mixture of aromatic hydrocarbon and alcohol (or alcohols), by the Friedel-Crafts reaction, or by introducing olefines into arylsulphonic acids. (B) The process is restricted to the use of similar derivatives of "bicyclic aromatic bodies." H. A. P.

**Improvements in liquid fuels.** E. I. DU PONT DE NEMOURS & Co., F. B. DOWNING, and H. W. WALKER (B.P. 398,219, 7.3.32).—An inhibitor for gum formation and discoloration in liquid fuels (*e.g.*, petrol) comprises an amine salt of a phenolic compound (other than a hydroxydiaryl) in the absence of dyestuffs. A list of suitable substances is given. H. S. G.

**Decolorisation of [mineral] oils with acid-activated earth and water.** W. S. BAYLIS and D. S. BELDEN, Assrs. to FILTROL Co. OF CALIFORNIA (U.S.P. 1,898,165, 21.2.33. Appl., 14.5.29).—A mixture is prepared of clay, H<sub>2</sub>O, and oil. Acid is added and the mixture added to the oil to be decolorised and heated to 121–399°. H<sub>2</sub>O is discharged from the mixture, additional clay is added, and the temp. brought to  $<$  that of the first heat-treatment. The mixture is agitated and finally the clay separated from the oil. H. S. G.

**Safely distilling and decolorising mineral oils.** D. S. BELDEN, Assr. to FILTROL Co. OF CALIFORNIA (U.S.P. 1,898,168, 21.2.33. Appl., 10.6.29).—A mixture of oil, *e.g.*, used automobile crank-case oil, dry-cleaners' gasoline, etc., and absorbent clay (*C*), *e.g.*, Filtrol, is heated in a flowing stream to  $\leq 260^\circ$  and emptied directly into a fractionating space (*A*), held under reduced pressure, below at least one layer of *C*, at the same time continuously injecting N<sub>2</sub> into the lower part of *A*. Heavy fractions and oily clay are removed from the bottom of *A*, and lighter fractions and inert gas are withdrawn from the top, separated, and the N<sub>2</sub> recycled. H. S. G.

**Removal of corrosive sulphur from hydrocarbon oils.** R. K. STRATFORD and W. P. DOOHAN, Assrs. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,904,173, 18.4.33. Appl., 21.5.28).—The oil is agitated for 15 min. at 100°/100 lb. per sq. in. with alkali solution, *e.g.*, aq. NaOH, containing a contact-promoting substance (0.1–5.0%), *e.g.*, naphthenic, sulphonic, or fatty acid soaps, rosin, Turkey-red oil. D. K. M.

**Treatment [hydrogenation] of hydrocarbons.** E. J. GOHR, Assr. to STANDARD-I. G. Co. (U.S.P. 1,904,133, 18.4.33. Appl., 2.2.31).—In the vapour-phase catalytic hydrogenation of petroleum oil the temp. of the reaction chamber is controlled by using as feed a mixture of fresh oil and heavy fractions from a previous operation. D. K. M.

**Manufacture of sweated paraffin from non-sweated paraffin and like substances.** R. MEIJER, Assr. to N. V. DE BATAAFSCHE PETROLEUM MAATS. (U.S.P. 1,903,929, 18.4.33. Appl., 29.7.30. Holl., 13.8.29).—Trays (*T*) filled with molten paraffin are carried by means of endless chains through a chilling tunnel (*C*) through which cold air is blown, with their perforated bottoms immersed in a trough of cold H<sub>2</sub>O, the paraffin thereby being congealed. The *T* are carried through a sweating tunnel (*S*) situated above *C*, through which hot air is blown, the sweated paraffin running from *T* being collected. In a chamber which is an extension of *S*, the remaining paraffin is melted off from *T* by means of steam. D. K. M.

**Lubricating oils.** W. HELMORE and E. W. J. MARDLES (B.P. 398,222, 9.3.32).—Sludge formation in lubricating oils (vegetable, mineral, or blends thereof) is considerably reduced by the addition of  $\geq 1\%$  of metallic Sn or of an oil-sol. compound of Sn (*e.g.*, the oleate, naphthyl, ricinoleate, Ph or Me iodide), with or without the further addition of PbEt<sub>4</sub> or Pb oleate. H. S. G.

**Recovery of solvents from mixtures [with mineral lubricating oils] containing the same.** F. X. GOVERS, Assr. to INDIAN REFINING Co. (U.S.P. 1,897,979, 14.2.33. Appl., 7.5.31).—A mixture of COMe<sub>2</sub> and C<sub>6</sub>H<sub>6</sub> with mineral lubricating oil is preheated, and further heated, if necessary, to 82° to remove the bulk of the solvents. The unvaporised portion is transferred to a still (*S*) maintained under vac. and heated to 171° by rapid circulation over and spraying upon heated surfaces within *S*. The vapours from the initial distillation and from *S* are passed to a condenser under pressure. The hot, substantially vapour-free condensate is subjected to cooling under still higher pressure during countercurrent flow at high velocity in heat-exchange relation with a cooling liquid. The stripped oil is withdrawn from *S* for use in preheating a fresh charge. H. S. G.

**Oil stills.** C. E. LUCKE, Assr. to BABCOCK & WILCOX Co. (U.S.P. 1,908,374–5, 9.5.33. Appl., 28.3.28).

**Oil cooler and filter.** K. E. LYMAN, Assr. to BORG-WARNER CORP. (U.S.P. 1,906,984, 2.5.33. Appl., 12.9.30).

**Cracking of petroleum vapours.** C. W. ANDREWS and R. D. ROGERS, Assrs. to C. P. T. DEVELOPMENT Co. (U.S.P. 1,907,029, 2.5.33. Appl., 3.3.30).

**Cracking of oils.** C. P. DUBBS, Assr. to UNIVERSAL OIL PRODUCTS Co. (U.S.P. 1,897,635, 14.2.33. Appl., 9.3.23. Renewed 19.12.29).

**Conversion of hydrocarbon oils.** H. R. SNOW, Assr. to STANDARD OIL Co. (U.S.P. 1,908,001, 9.5.33. Appl., 25.4.30).



**Treatment of hydrocarbons.** E. E. STEWART (U.S.P. 1,905,071, 25.4.33. Appl., 11.2.29).

**Apparatus for treating hydrocarbons.** J. C. BLACK, Assr. to GASOLINE PRODUCTS Co., INC. (U.S.P. 1,908,106, 9.5.33. Appl., 28.5.25).

**Apparatus for treating hydrocarbon oils.** O. C. BREWSTER and W. M. PERRY, Assrs. to STANDARD OIL Co. (U.S.P. 1,909,335, 16.5.33. Appl., 22.3.28).

**Distillation of hydrocarbon oils.** S. C. YEATON (U.S.P. 1,909,600, 16.5.33. Appl., 28.5.30).

**Distillation of petroleum oils.** A. A. MACCUBBIN and J. ZAVERTNIK, JUN., Assrs. to W. W. GRIMM (U.S.P. 1,907,328, 2.5.33. Appl., 15.12.28).

**Treatment of oil gas.** C. W. ANDREWS and A. J. BOYNTON, Assrs. to H. A. BRASSERT & Co. (U.S.P. 1,905,028, 25.4.33. Appl., 8.10.28).

**Division of petroleum bottoms.** G. B. COUBROUGH, Assr. to LUMMUS Co. (U.S.P. 1,905,156, 25.4.33. Appl., 8.3.32).

**Gas machine [for carburetting air with gaseous hydrocarbons].** R. E. WILSON, Assr. to STANDARD OIL Co. (U.S.P. 1,907,924, 9.5.33. Appl., 30.6.32).

**Mixing device [for hydrocarbon oil-reagent mixtures].** F. W. EBAUGH, Assr. to TEXAS Co. (U.S.P. 1,906,951, 2.5.33. Appl., 18.9.29).

**Separation of materials [coal and stone]. Removing acid constituents from gases.**—See I.  $C_2H_2$  chlorides. Alkyl sulphates. Ester purification. Lubricants.—See III. Bituminous concrete etc.—See IX. Steel apparatus [for oil cracking]. Corrosion-resistant coatings.—See X. Exterminating pests.—See XVI.

### III.—ORGANIC INTERMEDIATES.

**Thermal decomposition of methane from Sarmasel (Rumania).** C. CANDEA and J. KÜHN (Chim. et Ind., 1933, 29, Spec. No., 861—865).—The best catalyst for the conversion of  $CH_4$  into  $H_2$  and C at 800—1000° contained  $Fe_2O_3$ ,  $CaCO_3$ , NiO, and  $MnO_2$  in the proportions 10, 4, 2, and 1, respectively. The C has strong adsorbent properties. A. A. L.

**Dehydration of alcohol by absorbent and recuperated vapour.** G. DU BOIS (Chim. et Ind., 1933, 30, 535—536).—The original method of dehydration of EtOH by means of CaO was supplanted by azeotropic methods, but recent advances have tended to reintroduce absorbents both solid and liquid. The condensed and conc. distillate is treated in this way. An economical method of treating instead the vapour is as follows. If 94% EtOH is to be freed from  $H_2O$  the feed into the top of the column (A) is enriched to 98% (i.e., above the azeotropic point). The vapours are then dehydrated with absorbent in another vessel and returned to A by a compressor (B). The economy depends on the efficiency of C so that pressure drop should be kept at a min. Other applications are given. The method produces a particularly pure EtOH. C. I.

**Concentration of acetic acid.** B. VON TETMAJER (Chim. et Ind., 1933, 29, Spec. No., 866—867; cf. B., 1932, 55). A. A. L.

**Manufacture of ethylene oxide and ethylene glycol.** J. FRANÇON (Chim. et Ind., 1933, 29, Spec. No., 869—875).—A mixture (1:10) of  $C_2H_4$  and air is passed over a catalyst heated at 350° to give  $C_2H_4O$  (I) and  $CO_2$ .  $C_2H_4$  from coke-oven plants may be used, after pretreatment to remove impurities, and the residual  $C_2H_4$  may either be further oxidised to  $C_2H_4(OH)_2$  or converted into EtOH. A. A. L.

**Catalyst for production of butyl alcohol from ethyl alcohol.** B. N. DOLGOV and J. N. VOLNOV (J. Gen. Chem. Russ., 1933, 3, 313—318).—The process, consisting of the reactions  $EtOH \rightarrow C_2H_4 + H_2O$  (I) and  $EtOH + C_2H_4 \rightarrow BuOH$  (II), is complicated by the side-reactions  $EtOH \rightarrow CO + CH_4 + H_2$  (III) and  $2EtOH \rightarrow CO_2 + 3CH_4$  (IV).  $Al_2O_3$  catalyses reaction (III), NiO chiefly reactions (III) and (IV), and animal charcoal is a feeble catalyst of reactions (III) and (IV), which are largely suppressed at 450° in presence of a mixture of oxides of Fe, Al, and La, pptd. on C; with this catalyst 22% yields of BuOH, together with  $CH_2O$ ,  $COMe_2$ , olefines,  $MeCHO$ ,  $(C_5H_{11})_2O$ , and  $Bu_2O$ , are obtained. R. T.

**Catalytic production of camphene from turpentine.** V. E. TISCHTSCHENKO and G. A. RUDAKOV (J. Appl. Chem. Russ., 1933, 6, 691—704).—Boiling pinene, alone or in turpentine, is converted into camphene (I) (56—62% yields) and dipentene (10—25%) in presence of 0.5% of activated clay (II), prepared by boiling clay with 10% HCl during 6 hr. and drying the product at 125°; the action of (II) is analogous to that of acids. The duration of contact should be  $> 30$  min., as the (I) produced undergoes polymerisation. Bertram and Walbaum's method for determination of (I) cannot be applied to mixtures rich in pinene, 1.5—2% of which undergoes conversion into terpineol, and 14—18% into isobornyl acetates, which are hydrolysed together with bornyl acetate derived from (I). R. T.

**Removal of thiophen from benzene by catalytic hydrogenation.** I. R. N. PEASE and W. B. KEIGHTON, JUN. II. R. N. PEASE and W. C. MUNRO (Ind. Eng. Chem., 1933, 25, 1012—1013, 1013).—I.  $C_5H_5S$  is removed from  $C_6H_6$  by heating with  $H_2$  at 325° in presence of an equimol.  $CoS-MoO_3$  mixture as catalyst. Reaction at 200° has been examined in detail.

II.  $CoS-Cr_2O_3$ ,  $Cr_2O_3.xH_2O$ , and  $CoO-MoS_2$  are good catalysts, whilst Co treated with  $H_2S$  is inactive. F. R. S.

**Determination of mononitrotoluene.** K. LESNÍČENKO (Chim. et Ind., 1933, 29, Spec. No., 945—951).—Storm's results (B., 1912, 904) in nitrometer determinations with nitroglycerin, nitroglycol, and  $KNO_3$  to which small amounts of mono- (A), di- (B), and tri- (C) -nitrotoluene were added are confirmed; with B and C there was no loss of  $N_2$ , but with A the loss was proportional to the amount of  $NO_2$ -compound present. In the application of this to the determination of A, a standard mixed acid ( $H_2SO_4$  35,  $HNO_3$  15.3,  $H_2O$  49.7%; N 3.4%) was used, the residual N, as found by the nitrometer, deducted from the original N, giving the equiv. of A. W. J. W.

**Hydrogenation.**—See I. [Products from] waste sulphite liquor.—See II.  $C_2HCl_3$  and corrosion.—See X. Wetting-out etc. agents.—See XII.  $PrCO_2H$



and BuOH by fermentation. — See XVIII.  $C_6H_5Me(NO_2)_2$ . — See XXII.

See also A., Oct., 1006, Prep. of pure  $Me_3BO_3$  and  $Et_3BO_3$ . 1032, Oxidation of paraffins to fatty acids. 1039, Prep. of nitriles. 1042, Prep. of  $Ph_2$ . 1043, Prep. of alkylanilines containing *tert.*-alkyl groups. 1048, Synthesis of BzOH. 1053, Conversion of phthaloylnaphthalenes and naphthoyl-2-benzoic acids into benzantraquinones. 1082, Prep. of  $H_2C_2O_4$  from  $HCO_2H$ . Bacterial production of amyl alcohol. 1083, Fermentation of polyhydric alcohols by bacteria.

## PATENTS.

**Preparation of unsaturated hydrocarbons.** W. O. HERRMANN and E. BAUM, Assrs. to CONSORT. F. ELECTROCHEM. IND. (U.S.P. 1,898,301, 21.2.33. Appl., 30.7.27. Ger., 2.8.26).—Saturated hydrocarbons are passed over C or with (or without) insufficient  $O_2$  for complete combustion over catalysts at red heat to  $1000^\circ$  with a space velocity of approx. 160 litres per litre of reaction space.  $CH_4$  is thus converted into  $C_2H_4$  and  $C_2H_2$ .

H. A. P.

**Manufacture of acetylene chlorides.** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 397,961, 22.4.32).—Acetylenic cracked gases are treated with  $Cl_2$  at  $80-140^\circ$ , preferably  $100-130^\circ$ , whereby formation of HCl etc. is avoided.

C. H.

**Oxidation of organic substances. [Preparation of formaldehyde from acetaldehyde.]** W. O. HERRMANN and E. BAUM, Assrs. to CONSORT. F. ELEKTROCHEM. IND. (U.S.P. 1,864,541, 28.6.32. Appl., 1.5.25. Ger., 6.5.24).—A mixture of MeCHO and  $O_2$  is forced at high velocity through a capillary tube and impinges on a heated surface (at, e.g.,  $400^\circ$ ), the products being led back over the capillary for heat exchange. Nearly quant. yields of  $CH_2O$  are obtained.

C. H.

**Catalytic molecular association [polymerisation] of aldehydes.** A. O. JAEGER, Assr. to SELDEN CO. (U.S.P. 1,864,908, 28.6.32. Appl., 28.3.29).—Zeolites and other base-exchange substances catalyse aldolisation and crotonisation at  $80-200^\circ$ .

C. H.

**Manufacture of methylamine.** H. DREYFUS (B.P. 398,502-4, 9.3.32).—(A, c) HCN or (B) cyanides are reduced with  $H_2$  (A) in presence of cyanides of hydrogenating metals (Ni, Cu, Zn), with or without the metals themselves, (c) in presence of Cu, Zn, Co, Sn, or Ag, (B) in (aq.) solution or suspension free from other acid ions, in presence, if desired, of cyanides of hydrogenating metals. In each case a cyanide of an alkali or alkaline-earth metal may be present. The preferred temp. is (A, c)  $200-350^\circ$ , (B)  $70-95^\circ$  or, under pressure,  $150-200^\circ$ .

C. H.

**Manufacture of ethyl alcohol [from ethylene].** DISTILLERS CO., LTD., W. P. JOSHUA, H. M. STANLEY, and J. B. DYMÖCK (B.P. 396,724, 29.1.32. Cf. B.P. 392,289; B., 1933, 776).—For the hydration of  $C_2H_4$  a  $B_2O_3-H_3PO_4$  catalyst is used containing excess of  $H_3PO_4$ , e.g., 1 mol. of  $B_2O_3$  to 2.2 mols. of  $H_3PO_4$  at  $200-270^\circ/1-20$  atm.

VHOC

C. H.

**Foam-prevention agent.** L. J. CLAPSADLE, Assr. to CARBIDE & CARBON CHEMICALS CORP. (U.S.P. 1,899,610,

28.2.33. Appl., 6.1.31).—Foaming of aq. alcohols [ $C_2H_4(OH)_2$ ] is prevented by 0.01–0.1% of a neutral salt of an alkaline-earth metal [ $Ca(OAc)_2$ ]. H. A. P.

**Recovery of acetic acid from solutions obtained in cellulose derivative manufacture.** E. PRINCE, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,898,213; 21.2.33. Appl., 1.12.26. Fr., 26.2.26).—A mineral acid is added and the solution heated to convert cellulosic substances into  $H_2O$ -sol. degradation products before extraction of the AcOH.

H. A. P.

**Dehydration of acetic acid.** D. F. OTHMER, Assr. to EASTMAN KODAK CO. (U.S.P. 1,897,816, 14.2.33. Appl., 19.1.31).—Aq. AcOH is neutralised with  $N(CH_2CH_2OH)_3$  and the product distilled, preferably under reduced pressure.

H. A. P.

**Manufacture of aliphatic acids, anhydrides, and ketones.** H. DREYFUS (B.P. 396,770, 9.2.32).—Suitable salts of aliphatic acids are heated in an inert diluent which boils above the decomp. temp. of the salt. E.g., 25% AcOH is converted into  $Cu(OAc)_2 \cdot 5H_2O$ , which is then heated in petroleum of b.p.  $290-330^\circ$ , first at  $90^\circ$  to remove  $H_2O$ , then at  $280^\circ$  to give conc. AcOH.

C. H.

**Manufacture of mixed anhydrides of boric and organic acids.** HENKEL & Co., G.M.B.H. (B.P. 398,064, 7.12.32. Ger., 23.12.31).—A mixture of an org. acid (except AcOH) and  $H_3BO_3$  or  $B_2O_3$  is treated with an inorg. acid chloride or an org. acid anhydride ( $Ac_2O$ ). Examples are mixed anhydrides of boric and stearic (m.p.  $71^\circ$ ), lauric (b.p.  $180^\circ/2$  mm.), coconut-oil, benzoic (m.p.  $166^\circ$ ), and cinnamic (m.p.  $125-130^\circ$ ) acids.

C. H.

**Hydrogenation of carboxylic acid [to alcohols, and catalyst therefor].** E. I. DU PONT DE NEMOURS & Co. (B.P. 397,938, 7.3.32. U.S., 5.3.31).—A mixed chromite of Zn, Cd, and Cu is used as catalyst for reduction of aliphatic acids, preferably  $>C_8$ , to alcohols (and esters), e.g., at  $350-420^\circ/160-200$  atm. Examples are alcohols from lauric, butyric, acetic, ricinoleic, oleic, stearic, and coconut-oil acids. Olefinic linkings may be subsequently reduced.

C. H.

**Manufacture of esters [from lower aliphatic acids and olefines].** E. H. STRANGE and T. KANE (B.P. 398,527, 8.3.32).—In the condensation of olefines with aliphatic acids  $<C_6$  in presence of catalysts (chlorides, bromides, phosphates, or sulphates of Zn, Al, Hg, Cu, Cd, Bi, or Sb;  $H_2SO_4$ ,  $H_3PO_4$ ) at temp. above the b.p. of the ester, the ester is removed continuously as vapour. The prep. of  $PrOAc$  (15–20% conversion) is described.

C. H.

**Manufacture of vinyl compounds.** H. DREYFUS (B.P. 398,173, 1.2.32. Cf. B.P. 385,978; B., 1933, 217).—A vinyl halide is condensed with compounds containing replaceable H and  $\leq 1$  hydrophile group. Glycol monovinyl ether, b.p.  $110-150^\circ$ , and vinyl H succinate so prepared yield polymerides readily dispersed in  $H_2O$ .

C. H.

**Manufacture of polymerised vinyl alcohol.** C. R. NOLLER, Assr. to EASTMAN KODAK CO. (U.S.P. 1,897,856, 14.2.33. Appl., 6.2.29).—The hydrolysis of polyvinyl acetate is carried out in presence of sufficient  $H_2O$  to



prevent pptn. of  $(\text{CH}_2\text{CH}(\text{OH}))_n$ , and the product is pptd. with EtOH. Easier filtration is claimed.

H. A. P.

**Formation of alkyl sulphates.** S. R. MERLEY, ASSR. to DOHERTY RESEARCH Co. (U.S.P. 1,897,812, 14.2.33. Appl., 11.8.26).—The gases from cracked petroleum are passed into conc.  $\text{H}_2\text{SO}_4$  ( $d$  1.84) at  $-15^\circ$  until the  $d$  falls to  $< 1.263$ , and the acid liquor thus obtained is treated with the liquid products of cracking, still at  $-15^\circ$ . The solution of alkyl sulphates (mainly  $\text{Pr}^i\text{HSO}_4$ ) in  $\text{H}_2\text{SO}_4$  thus obtained is free from org. sulphonic acids, and the  $\text{H}_2\text{SO}_4$  recovered by hydrolysis may be re-used after concn.

H. A. P.

#### Manufacture of [phthalic] esters of butyl alcohol.

R. B. CROWELL and S. R. EBE, ASSRS. to AMER. SOLVENTS & CHEM. CORP. (U.S.P. 1,864,893, 28.6.32. Appl., 24.9.28).—Excess BuOH and phthalic anhydride are heated with  $\text{H}_2\text{SO}_4$  at  $82-87^\circ$  and the binary mixture of  $\text{H}_2\text{O}$  and BuOH is distilled off first at 250 mm. and finally at 100 mm., leaving Bu phthalate in 97% yield.

C. H.

**Acetylation process.** F. O. RICE, ASSR. to ROHM & HAAS Co. (U.S.P. 1,898,687, 21.2.33. Appl., 24.10.31).—The substance to be acetylated (*e.g.*,  $\text{AcOH} \rightarrow \text{Ac}_2\text{O}$ ) in a dispersed form is passed in countercurrent to keten (or "a keten") at a temp.  $>$  its b.p. but  $<$  the b.p. of the Ac derivative ( $100-300^\circ$ ).

H. A. P.

**Ester purification.** S. R. MERLEY, ASSR. to DOHERTY RESEARCH Co. (U.S.P. 1,898,737, 21.2.33. Appl., 13.6.28).—Esters obtained from olefines ( $\text{Pr}^i\text{OAc}$ ) are freed from small amounts of more easily decomposed impurities ( $\text{Bu}^i\text{OAc}$ ) by heating with an aq. mineral acid containing the acid of the ester and separation of the undecomposed ester by distillation.

H. A. P.

**Production of thiourea.** R. V. HEUSER, ASSR. to AMER. CYANAMID Co. (U.S.P. 1,889,959, 6.12.32. Appl., 9.2.31).—A mixture of  $\text{P}_2\text{S}_5$  and dil. aq.  $\text{NH}_3$  is autoclaved at  $100-120^\circ$  to produce  $(\text{NH}_4)_2\text{S}$  and  $(\text{NH}_4)_2\text{HPO}_4$  and to the cold solution is added the filtered aq. extract of  $\text{CaCN}_2$ . The mixture is heated to expel  $\text{NH}_3$ , the  $\text{Ca}_3\text{PO}_4$  removed by filtration, and the solution cooled to recover  $\text{CS}(\text{NH}_2)_2$ .

A. R. P.

**Manufacture of reaction products of keten.** CARBIDE & CARBON CHEMICALS CORP., ASSEES. of G. H. LAW (B.P. 397,025, 7.2.33. U.S., 16.2.32).—Gases containing  $\text{CH}_2\text{:CO}$ , *e.g.*, pyrolysed  $\text{COMe}_2$ , are scrubbed with  $\text{COMe}_2$  or other inert solvent, *e.g.*, in countercurrent at  $35-55^\circ$ ; the  $\text{CH}_2\text{:CO}$  may be dissolved out from the scrubbed gas by a suitable solvent, *e.g.*,  $\text{COMe}_2$  at  $< 0^\circ$ , or the gas may be passed into a reactant at  $30-40^\circ$ . (Cf. B.P. 377,574; B., 1932, 973.)

C. H.

**Manufacture of condensation products [glycerol cyclic ethers].** H. DREYFUS (B.P. 396,761, 5.1.32).—A polyhydric alcohol ether or ester having  $\leq 2$  OH groups is condensed with the same or a similar derivative, or with a glycol or other polyhydric alcohol, or with an aliphatic aldehyde, with or without  $\text{H}_2\text{SO}_4$ ,  $\text{HCl}$ ,  $\text{CaCl}_2$ , or other condensing agent. Ether or ester groups may be hydrolysed during or after reaction, and condensation may be continued after hydrolysis until all OH groups are etherified. Examples are: monoacetin with glycerol

mono-Me ether; erythritol diacetate with  $\text{MeCHO}$ . The products are solvents, plasticisers, etc.

C. H.

**Production of highly polymerised aliphatic alcohols [lubricants and softeners].** K. NOACK (B.P. 398,474, 6.2.32).—Explosion-proof lubricants suitable for  $\text{O}_2$  cylinders, welding-burners, and bearings are made by condensation of glycerol, or other polyhydric alcohol having  $\leq 3$  OH groups, at  $250-275^\circ$  in presence of heavy-metal salts ( $\text{HgCl}_2$ ,  $\text{HgSO}_4$ ,  $\text{Cu}_2\text{Cl}_2$ ,  $\text{FeCl}_3$ ),  $\text{H}_2\text{O}$  being removed by a current of inert gas as produced. The product is diluted with  $\text{H}_2\text{O}$  before use, with addition, if desired, of hygroscopic substances ( $\text{MgCl}_2$ , lactates) and tragacanth, talcum, etc. to produce ointment consistency.

C. H.

**Manufacture of [sulphonated] derivatives of unsaturated fatty alcohols [wetting and cleansing agents].** DEUTS. HYDRIERWERKE A.-G. (B.P. 396,774, 11.2.32. Ger., 11.2.31).—An acylated unsaturated alcohol  $> \text{C}_7$  (*e.g.*, oleyl acetate) is treated with  $\text{ClSO}_3\text{H}$ , oleum, or other sulphonating agent at  $20-30^\circ$ , whereby *C*-sulphonic acids are formed to the exclusion of sulphuric esters. Preferably the acyl group is thereafter removed and further  $\text{SO}_3\text{H}$  groups are introduced at  $> 35^\circ$ .

C. H.

**[Manufacture of] detergent agents.** IMPERIAL CHEM. INDUSTRIES, LTD., A. W. BALDWIN, and H. M. BUNBURY (B.P. 398,818, 9.2.32).—Batyl, chimyl, and/or selachyl alcohol, or other monoalkyl ether of glycerol having  $\text{C}_{19}-\text{C}_{21}$ , is treated with a sulphonating agent, particularly with  $\text{C}_5\text{H}_5\text{N} \cdot \text{SO}_3$  or  $\text{Na}_2\text{S}_2\text{O}_7$  and  $\text{C}_5\text{H}_5\text{N}$ .

C. H.

**Manufacture of sulphonic acids of the terpene series [wetting, cleansing, and emulsifying agents].** SOC. CHEM. IND. IN BASLE (B.P. 398,086, 20.1.33. Switz., 21.1.32).—Terpineol is sulphonated, *e.g.*, with  $\text{ClSO}_3\text{H}$  or oleum at  $0^\circ$  to  $-10^\circ$ .

C. H.

**Manufacture of cyclohexylcyclohexanol and homologues thereof.** HOWARDS & SONS, LTD., J. W. BLAGDEN, and G. C. H. CLARK (B.P. 397,883, 1.2.32).—*cyclo*Hexylidenecyclohexanone or a homologue is hydrogenated to the corresponding *cyclo*hexylcyclohexanol in one operation by the use of a non-noble metal catalyst ( $\text{Ni}$  with  $\text{MgO}$ ). Products described are  $\text{C}_6\text{H}_{11} \cdot \text{C}_6\text{H}_{10} \cdot \text{OH}$ , b.p.  $264^\circ$ , and its  $\text{Me}_2$  derivative (mixed isomerides), b.p.  $265-280^\circ$ .

C. H.

**Preparation of citrylideneallylacetone.** H. BARBIER, ASSR. to GIVAUDAN-DELAUNAY, INC. (U.S.P. 1,898,075, 21.2.33. Appl., 18.9.30).—*Citrylideneallyl*-acetone, b.p.  $140-150^\circ/3$  mm., is prepared from citral and  $\text{CH}_2\text{:CH} \cdot \text{CH}_2\text{Ac}$  (I) in presence of alkaline catalysts, and converted by acids into *cyclocitrylideneallylacetone*, b.p.  $130^\circ/3$  mm., also obtained from *cyclocitral* and (I).

H. A. P.

**Reduction of aromatic hydroxy-compounds [to hydrocarbons].** J. Y. JOHNSON. From I. G. FARBERIND. A.-G. (B.P. 397,901, 27.2.32).—In the catalytic hydrogenation of phenols to  $\text{C}_6\text{H}_6$  etc. the time of contact at  $400-500^\circ/150-250$  atm. is reduced so that 10–40% of the phenol remains unconverted and is recirculated. Preferred catalysts are sulphides or selenides of Mo, W, or V.

C. H.



**Stabilisation of chlorinated hydrocarbons.** ROESSLER & HASSLACHER CHEM. CO. (B.P. 397,914—5, 3.3.32. U.S., 3.3.31).—Up to 0.1% of (A) amylene, (B) arylamines or phenols (e.g.,  $\text{NH}_2\text{Ph}$ , *o*- and *p*- $\text{NH}_2\cdot\text{C}_6\text{H}_4\text{Me}$ , diphenylguanidine,  $\text{PhOH}$ , cresols, pyrocatechol, thymol, quinol, pyrogallol, resorcinol, *o*- $\text{NH}_2\cdot\text{C}_6\text{H}_4\cdot\text{OH}$ , or  $\alpha\text{-C}_{10}\text{H}_7\cdot\text{OH}$ ), are added to  $\text{CH}_2\text{Cl}_2$ ,  $\text{C}_2\text{H}_2\text{Cl}_2$ ,  $\text{C}_2\text{HCl}_3$ ,  $\text{C}_2\text{Cl}_6$ ,  $\text{C}_2\text{H}_2\text{Cl}_4$ , etc. C. H.

**Production of aromatic alcohols.** L. and I. VALIK (B.P. 398,136, 23.5.33. U.S., 1.6.32).—In the condensation of aromatic hydrocarbons with ethylene oxide in presence of  $\text{AlCl}_3$  etc. the oxide is diluted with  $\text{N}_2$ ,  $\text{CO}_2$ , or other inert gas. Preferably reaction is effected at 5–10° and an amine (triethanolamine) is added to neutralise  $\text{HCl}$ . The phenylethyl alcohol, or other product, may be purified by treatment with aq.  $\text{KMnO}_4$  followed by aq.  $\text{NaHSO}_3$ , or by dissolution in white wax or mineral oil. C. H.

**Manufacture of  $\beta$ -phenylethyl alcohol.** P. SCHORIGIN, V. I. ISAGULJANZ, A. R. GUSSEVA, K. S. PALIAKOV, and V. P. OSSIPOVA (B.P. 398,561, 21.3.32).— $\text{PhCl}$  is heated at 150–160° with  $\text{Mg}$  in absence of solvent [ $\text{PhMgCl}$  (I) being added to catalyse the reaction, if desired], and the resulting (I) is treated in  $\text{C}_6\text{H}_6$  with ethylene halohydrin or oxide. C. H.

**Production of nuclear-substituted phenyl-aminoalkylcarbinols.** M. OBERLIN (U.S.P. 1,898,258, 21.2.33. Appl., 24.9.30. Ger., 21.10.29).— $\text{OH}\cdot\text{CHPh}\cdot\text{CH}_2\cdot\text{NH}_2$  and its *N*-Me derivatives and the *N*-Me derivatives of  $\text{OH}\cdot\text{CHPh}\cdot\text{CHMe}\cdot\text{NH}_2$  are nitrated at < 10–30° ( $\text{HNO}_3$ , *d* 1.4 or  $\text{HNO}_3\text{-H}_2\text{SO}_4$ ), and the products reduced (in most cases with  $\text{H}_2\text{-Pd}$ ). The following hydrochlorides are described: (?) *dl*-, m.p. 234–240° [ $\beta$ -*N*-Me<sub>2</sub> derivative, m.p. 205–210° (decomp.)], and 1- $\beta$ -amino- $\alpha$ -*p*-nitrophenylpropanol, m.p. 138–139°; (?) *dl*-, m.p. 192–193° (decomp.) [ $\beta$ -*N*-Me<sub>2</sub> derivative, 188–190° (decomp.)], 1-, m.p. 164–166° (decomp.),  $[\alpha]_D^{20}$  –33° in  $\text{H}_2\text{O}$ , and *d*- $\beta$ -amino- $\alpha$ -*p*-aminophenylpropanol, m.p. 171–173° (decomp.);  $\beta$ -methylamino- $\alpha$ -*p*-nitro-, m.p. 162–165°, and  $\alpha$ -*p*-amino-phenylethanol, m.p. 160–162°. H. A. P.

**Production of valuable [alkyl] derivatives [germicides] from phenols.** STANDARD OIL DEVELOPMENT Co., Assees. of H. E. BUC (B.P. 398,218, 7.3.32. U.S., 12.3.31).—An unsulphonated phenol (I) is condensed with an aliphatic or alicyclic olefine (II) >  $\text{C}_2$  in conc.  $\text{H}_2\text{SO}_4$  at 15–70°, the proportion of  $\text{H}_2\text{SO}_4$  being < 1 mol. per mol. of (I) or (II) reacting. Any  $\text{SO}_3\text{H}$  groups are removed by hydrolysis. Homologous phenols, accompanied by phenol ethers, are thus obtained from:  $\text{PhOH}$  with  $\Delta^8$ -pentene (b.p. 238–244°),  $\Delta^8$ -hexene (b.p. 250–260°), *sec*-heptenes (b.p. 270–285°), and 2 mols. of  $\text{C}_3\text{H}_6$  (b.p. 231–242°); technical cresol with  $\Delta^8$ -hexene (b.p. 259–263°), and  $\text{CMe}_2\cdot\text{CHMe}$  (b.p. 207–251°); resorcinol with  $\Delta^8$ -hexene (b.p. 155–180°/1 mm.). *sec*-Hexylresorcinol is a more powerful germicide etc. than the *n*-compound. C. H.

**Manufacture of optically active 1-monohydroxy-phenyl-2-aminopropanols-1 [ $\beta$ -amino- $\alpha$ -hydroxy-phenylpropyl alcohols].** I. G. FARBERIND, A.-G. (B.P. 396,951, 19.9.32. Ger., 19.9.31).—The *dl*-com-

pounds are resolved by means of *d*-tartaric acid. For the  $\beta$ - $\text{NHMe}\cdot\alpha\text{-m-C}_6\text{H}_4\cdot\text{OH}$ -compound *dB,dA*, m.p. 144–145°, is the less sol. in  $\text{EtOH}$  and gives *dB*, m.p. 216–217°,  $[\alpha]_D^{20}$  +27.33°; *lB* is obtained from the mother-liquors. For the  $\beta$ - $\text{NH}_2\cdot\alpha\text{-m-C}_6\text{H}_4\cdot\text{OH}$ -compound, *lB,dA*, m.p. 176–177°, is the less sol. in  $\text{MeOH}$  and gives *lB*, m.p. 190°,  $[\alpha]_D^{20}$  –21.66°. C. H.

**Manufacture of mixed phenyl alkyl esters [plasticisers].** M. N. DVORNIKOFF, ASSR. to MONSANTO CHEM. WORKS (U.S.P. 1,899,919, 28.2.33. Appl., 15.10.31).—The alkyl H ester of a polycarboxylic acid is converted into its chloride by  $\text{PCl}_3$ ,  $\text{SOCl}_2$ , etc., which is then allowed to react with a phenol. *Ph Me phthalate*, b.p. 191°/5 mm., *Ph Et phthalate*, b.p. 195°/4 mm., and *Ph Me succinate*, b.p. 133°/10 mm., are thus prepared. H. A. P.

**[C]-Dialkylresorcinols [germicides].** W. G. CHRISTIANSEN and S. E. HARRIS, ASSRS. to E. R. SQUIBB & SONS (U.S.P. 1,897,188, 14.2.33. Appl., 7.7.32).—*C*-Acyl derivatives of *C*-alkylresorcinols, produced by the Gattermann or Fries reaction, are reduced by Clemmensen's method. *C*-*n*-Butylresorcinol mono-acetate, b.p. 147–150°/2 mm., and -propionate, b.p. 161–165°/2 mm.; *C*-*n*-butylresacetophenone, b.p. 164–166°/3 mm.; and *C*-*n*-propionyl-*n*-butyl-, -methyl-*n*-amyl-, b.p. 165–170°/2.5 mm., -ethyl-*n*-butyl-, b.p. 150–151°/2.5 mm., and -*n*-propyl-*n*-butyl-resorcinol, b.p. 153–160°/3 mm., are described. H. A. P.

**Production of organic amines.** G. T. MORGAN and D. D. PRATT (B.P. 396,760, 11.12.31).—An alcohol or a monohydric phenol is heated with an  $\text{NH}_4$  or amine salt, e.g., at 300–400°/100–200 atm. Examples are amines from:  $\text{EtOH}$ , *m*-cresol, or *m*-5-xyleneol with  $\text{NH}_4\text{Cl}$ ; *m*-5-xyleneol with  $\text{NHMe}_2\cdot\text{HCl}$  (product, b.p. 230–260°). C. H.

**Manufacture of aldehyde-amine reaction products [vulcanisation accelerators].** C. O. NORTH, ASSR. to RUBBER SERVICE LABORATORIES CO. (U.S.P. 1,897,210, 14.2.33. Appl., 29.3.26).— $\text{PrCHO}$  (approx. 3 equivs.) and  $\text{NH}_2\text{Ph}$  (1 equiv.) are heated together under reflux, preferably with removal of the  $\text{H}_2\text{O}$  formed, and the product is heated at 100–145° for 2–20 hr. H. A. P.

**Manufacture of tetrazo compounds [from *m*- or *p*-phenylenediamine].** I. G. FARBERIND, A.-G. (B.P. 397,034, 24.2.33. Ger., 24.2.32).—The diamines are tetrazotised smoothly by means of  $\text{NaNO}_2$  and  $\text{HBF}_4$ . Examples are *m*- and *p*- $\text{C}_6\text{H}_4(\text{NH}_2)_2$ , 2:5-dichloro- and 2:5-dimethoxy-*p*-phenylenediamines. The bisdiazonium borofluorides are pptd., and are sol. in  $\text{H}_2\text{O}$ . C. H.

**Continuous preparation of disubstituted guanidines.** W. P. TER HORST, ASSR. to RUBBER SERVICE LABORATORIES CO. (U.S.P. 1,897,220, 14.2.33. Appl., 7.5.28).—An apparatus is described in which  $\text{NH}_2\text{Ph}$ , *o*- $\text{NH}_2\cdot\text{C}_6\text{H}_4\text{Me}$ , or other arylamine is passed in counter-current to  $\text{CNCl}$  containing < 1.7%  $\text{H}_2\text{O}$  at 90–115°, the resulting diarylguanidine hydrochloride dissolved in  $\text{H}_2\text{O}$ , freed from excess amine by extraction with an inert solvent, and the base pptd. by alkali. H. A. P.

**Purification of phthalic anhydride.** P. C. BOWERS, ASSR. to E. I. DU PONT DE NEMOURS & CO. (U.S.P.



1,897,110, 14.2.33. Appl., 24.8.29).—Crude phthalic anhydride is treated with 0.25–1 wt.-% of  $\text{H}_2\text{SO}_4$ , alkali H sulphates, or org. sulphonic acids before sublimation or distillation. H. A. P.

**Separation or purification of chlorinated derivatives of phthalic acids.** E. G. BECKETT, C. SHAW, W. E. STEPHEN, G. C. SEMPLE, R. F. THOMSON, and IMPERIAL CHEM. INDUSTRIES, LTD. (B.P. 396,735, 9.2.32).—50–100%  $\text{H}_2\text{SO}_4$  is used for the preferential conversion of anhydrides into acids or of acids into anhydrides, anhydride being subsequently extracted with a suitable solvent (PhMe). Anhydrides of phthalic acid and its 4-Cl- and 4 : 5- $\text{Cl}_2$ -derivatives are converted into acids by 98–100%  $\text{H}_2\text{SO}_4$ ; 3-Cl-, 3 : 4- and 3 : 5- $\text{Cl}_2$ -derivatives by 55–85%  $\text{H}_2\text{SO}_4$ ; 3 : 6- $\text{Cl}_2$ - and  $\text{Cl}_4$ -derivatives only by < 50%  $\text{H}_2\text{SO}_4$ . The reverse reaction occurs with  $\text{H}_2\text{SO}_4$  of higher concn. The unisolated product of dichlorination of phthalic anhydride is treated, e.g., with 98–99%  $\text{H}_2\text{SO}_4$ , the 3 : 4- and 3 : 6- $\text{Cl}_2$ -anhydrides are dissolved in PhMe (leaving 4 : 5- $\text{Cl}_2$ -acid), and the solution treated with 56%  $\text{H}_2\text{SO}_4$ ; 3 : 4- $\text{Cl}_2$ -acid is filtered off, and 3 : 6- $\text{Cl}_2$ -anhydride recovered from the filtrate. C. H.

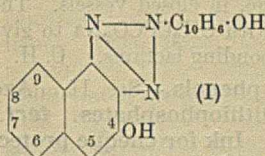
**Purification of diphenyl oxide [ether].** E. C. BRITTON and W. R. REED, Assrs. to Dow Chem. Co. (U.S.P. 1,899,257, 28.2.33. Appl., 3.3.32).— $\text{Ph}_2\text{O}$  is dissolved in 0.4 of its vol. of MeOH at 23–30°, the solution cooled to 10°, and the deposited crystals are distilled at 25–75 mm. H. A. P.

**Conservation of technical and pharmaceutical preparations and other substances liable to attack by micro-organisms.** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 396,737, 9.2.32).—Halogen-free monohydroxydiarylmethanes, or their salts with inorg. or org. bases, are used; e.g., *o*-hydroxydiphenylmethane is added to glue, or with glyceryl diacetate to gelatin; the *p*-OH-compound in "ethanolamine" (commercial) to casein. C. H.

**Manufacture of intermediate products and [azo] dyes therefrom.** SOC. CHEM. IND. IN BASLE (B.P. 399,097, 27.2.33. Switz., 25.2.32).—1 : 4-Diamino-8-naphthol-6-sulphonic acid, obtained by reduction of  $\text{NH}_2\text{Ph} \rightarrow$  J-acid, is diacylated and coupled with suitable diazo compounds. Examples are:  $\text{Ac}_2$  compound  $\leftarrow \text{NH}_2\text{Ph}$  (red) or *o*-anisidine (violet); di-*m*- or -*p*-aminobenzoyl compound  $\leftarrow$  2 : 4 : 8-naphthylaminedisulphonic acid (blue; green on development with pyrazolones etc.), or  $\leftarrow$  2 : 5-naphthylaminesulphonic acid  $\leftarrow$  *O*-acetyl-J-acid, hydrolysed (blue; green with suitable developments), or  $\leftarrow \alpha\text{-C}_{10}\text{H}_7\text{-NH}_2 \leftarrow$  *O*-*p*-toluenesulphonyl-H-acid; di-*m*-nitrobenzoyl compound  $\leftarrow$  3-nitro-*p*-toluidine on the fibre (lilac), or  $\leftarrow$  4-amino-4'-ethoxydiphenylamine (blue-grey). C. H.

**Manufacture of 2-(3'-hydroxy-2'-naphthyl)-4-hydroxy-6 : 7-benzo- $\phi$ -aziminobenzenes [4-hydroxy-*N*-(3-hydroxy-2-naphthyl)- $\alpha$ -naphthiminazoles] and azo dyes therefrom [ice colours and pigments].** I. G. FARBENIND. A.-G. (B.P. 396,741, 9.2.33. Ger., 9.2.32).—Dyes from an *O*-acyl- or *O*-alkyl-2 : 3-amino-naphthol, having free 1 and 4 positions, diazotised and coupled with the same or a similar aminonaphthol, are converted into corresponding azimino-compounds and

the *O*-substituents are removed. The products (I) have



good affinity for cotton and may be coupled on the fibre, or in substance, with diazo components free from solubilising groups. *O*-*p*-Toluenesulphonyl-2 : 3-amino-naphthol (II)  $\rightarrow$  (II), m.p. 198–199°,

gives (I), m.p. 282–283°, also obtained from 2 : 3-amino-naphthyl Me ether (III)  $\rightarrow$  (III), m.p. 196°. 8 : 7'- $\text{Br}_2$ - (m.p. > 320°), 8 : 7'-( $\text{OMe}$ )<sub>2</sub>- (m.p. 265–267°), and 8-Br- (m.p. 296–297°) derivatives of (I) are described. Examples of couplings include (I) with diazotised 4-chloro-*o*-toluidine (red-orange), 4-amino-3 : 2'-dimethylazobenzene (garret), 4-benzamido-2 : 5-dimethoxyaniline (red-violet), *p*-nitroaniline  $\rightarrow$  2 : 5-dimethoxyaniline (blue-black). The 8 : 7'-( $\text{OMe}$ )<sub>2</sub>-compound gives brown dyes. C. H.

**Manufacture of "silver salt" [sodium anthraquinone-2-sulphonate].** A. O. JAEGER and H. J. WEST, Assrs. to SELDEN RESEARCH & ENG. CORP. (U.S.P. 1,899,507, 28.2.33. Appl., 28.3.31).—Crude anthraquinone, obtained by catalytic air-oxidation of anthracene, and condensed on surfaces at the temp. of saturated steam/80–140 lb. per sq. in., is used as raw material for sulphonation. H. A. P.

**Manufacture of intermediate products and dyes [indigoids with anthraquinonylamide groups].** SOC. CHEM. IND. IN BASLE (B.P. 398,053, 11.11.32. Switz., 21. and 28.11.31).—A thiol- or amino-naphthoic acid is converted into the carboxynaphth-indoxyl, -thioindoxyl, -isatin, or -thioisatin, which may then be condensed with an aminoanthraquinone and converted by oxidation or by condensation with a suitable  $\text{CH}_2$ -compound into an indigo or thioindigo carrying a  $\cdot\text{CO}\cdot\text{NH}\cdot\text{Aq}$  group (Aq = anthraquinonyl). Examples include: 3-carboxy-2-naphthylthioglycollic acid, converted *via* the acid chloride into  $\beta$ -naphthathioindoxyl-4-carboxylic acid, m.p. > 250°, and thence into the thioisatin  $\alpha$ -dimethylaminoanil, m.p. > 350°, and  $\beta$ -naphthathioisatin-4-carboxylic acid, m.p. 296–297°, which is condensed with  $\beta\beta$ -naphthathioisatin  $\alpha$ -dimethylaminoanil and 1-amino-5-benzamidoanthraquinone (I) (olive-green vat dye), or with acenaphthaquinone and 1-amino-4-benzamidoanthraquinone (II) (brick-red); 4-carboxy-2-naphthylthioglycollic acid, m.p. 184°, converted into  $\beta$ -naphthathioindoxyl-5-carboxylic acid, m.p. > 230°, and condensed with  $\beta$ -naphthathioisatin  $\alpha$ -dimethylaminoanil and (I) (yellow-brown) or (II) (pure brown);  $\beta$ -naphthathioindoxyl-6-carboxylic acid, m.p. > 240°, oxidised and condensed with (II) (red-brown); 2-amino-3-naphthoic acid condensed with  $\text{CHBr}(\text{CO}_2\text{Et})_2$  and heated to give Et 4-carboxy- $\beta$ -naphthindoxyl-2-carboxylate, m.p. > 250°;  $\beta\beta$ -naphthathioindoxyl-9-carboxylic acid, m.p. 174–175°, from 1-carboxy-2-naphthylthioglycollic acid. C. H.

**[Manufacture of] mercapto- [thiol]-compounds of the anthraquinone series.** IMPERIAL CHEM. INDUSTRIES, LTD., F. LODGE, and C. H. LUMSDEN (B.P. 398,162, 29.1.32).—1-Amino-2 : 5- and -2 : 8-dithiolanthraquinones are obtained by treating 1-aminoanthraquinone-5- and -8-sulphonic acids with hot alkali



sulphide, or 1-chloro-5- and -8-nitroanthraquinones with hot alcoholic alkali H sulphide, in closed vessels. The products may be condensed with  $\text{CH}_2\text{Cl}\cdot\text{CO}_2\text{Na}$  to give thioglycolic acids and corresponding lactams. C. H.

**C<sub>2</sub>H<sub>2</sub>. Purification of phenols.**—See II. **Azo intermediates.**—See IV. **Dithiophosphates. tert.-Org. phosphates.**—See VII. **Ink for cheque protection. Blue dye in writing [inks]. Coloured inks.**—See XIII. **Tanning agent.**—See XV. **Distillable spirits by fermentation.**—See XVIII.

#### IV.—DYESTUFFS.

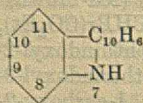
See A., Oct., 1045, **Synthesis of fast-violet B base and derivatives.** 1054, **Synthesis of naphthastaryl.**

##### PATENTS.

**Manufacture of nitrogenous derivatives of the anthraquinone series.** SOC. CHEM. IND. IN BASLE (B.P. 396,976, 8.11.32. Switz., 22.12.31).—Triamino-hydroxyanthraquinones are obtained by heating leuco-1:4:5:8-tetrahydroxy- or -1:4-diamino-5:8-dihydroxy-anthraquinones, or an *N*-derivative of the latter, with aq.  $\text{NH}_3$  or monoalkylamine at  $> 100^\circ$  in presence of a little reducing agent ( $\text{Na}_2\text{S}_2\text{O}_4$ ); the leuco-compounds may be produced *in situ*. The products are oxidised, and may be sulphonated. 4:5:8-Triamino-1-hydroxyanthraquinone (blue on acetate silk) and its *N*-methylated and *N*- $\beta$ -hydroxyethylated derivatives are described. C. H.

**[Manufacture of] anthracene and anthraquinone [thioindigoid] dyes.** IMPERIAL CHEM. INDUSTRIES, LTD., F. LODGE, and C. H. LUMSDEN (B.P. 398,161, 29.1.32).— $\alpha$ -Anthracyl- or  $\alpha$ -anthraquinonyl-thioglycolic acids carrying in 5 or 8 positions a Cl-, Br-,  $\text{NO}_2$ -,  $\text{SO}_3\text{H}$ -,  $\text{CO}_2\text{H}$ -, OH-, an  $\text{NH}_2$ -, or a substituted OH- or  $\text{NH}_2$ -group are cyclised with  $\text{ClSO}_3\text{H}$ , or  $\text{ClSO}_3\text{H}$  in  $\text{H}_2\text{SO}_4$ , oxidation to the thioindigo occurring simultaneously. Examples are: lactam of 1-aminoanthraquinylene-2:5-dithioglycolic acid (pure brown vat dye) and the corresponding anthracene (brown), and the 2:8-compound (brown); 5- $\text{NH}_2$ - (red-brown), 5-Cl- (brown), 5-*o*-carboxyanilino- (purple-brown), and 5- $\text{SO}_3\text{H}$ - (brown acid dye) -derivatives of 1-anthraquinonyl-thioglycolic acid. C. H.

**Manufacture of azo dyes and intermediate products [ice colours and pigments].** A. CARPMAEL. From I. G. FARBENIND. A.-G. (B.P. 398,516, 15.3.32).—9- or 10-Hydroxynaphthacarbazoles are carboxylated, and the arylamides of the OH-acids are used as coupling components. 9-Hydroxy- $\beta$ -naphthacarbazole, m.p.  $195^\circ$ , from the 9-sulphonic acid, gives the 9-hydroxy-10-carboxylic acid, m.p.  $245^\circ$  (decomp.) (*m*-chloroanilide, m.p.  $273^\circ$ ; *p*-chloroanilide, m.p.  $292^\circ$ ; *o*-toluidide, m.p.  $254^\circ$ ). 9-Hydroxy- $\alpha$ -naphthacarbazole, m.p.  $241^\circ$ , from the 9-OMe-compound, gives the 9-hydroxy-10-carboxylic acid, m.p.  $241^\circ$  (decomp.) (anilide, m.p.  $251^\circ$ ; *o*-, *m*-, and *p*-chloroanilides, m.p.  $237^\circ$ ,  $249^\circ$ , and  $257^\circ$ , respectively; *p*-aniside, m.p.  $246^\circ$ ; 4- and 5-chloro-*o*-anisides, m.p.  $246^\circ$  and  $248^\circ$ , respectively; 4- and 5-chloro-*o*-toluidides, m.p.  $243^\circ$  and  $248^\circ$ , respectively; 3-carbazolylamide, m.p.  $> 300^\circ$ ).



The arylamides have good affinity for cotton and give brown ice colours. C. H.

**Manufacture of o-[hydr]oxyazo dyes [chrome dyes for wool].** W. W. GROVES. From I. G. FARBENIND. A.-G. (B.P. 396,895, 23.5. and 26.10.32).—An *o*-aminophenolmonosulphonic acid is diazotised and coupled with a benzo-2:4-dihydroxyquinoline. Examples are: 4-nitro- or 4-chloro-*o*-aminophenol-6-sulphonic acid  $\rightarrow$  2:4-dihydroxy- $\alpha$ -naphthaquinoline or 1:3-dihydroxy- $\beta$ -naphthaquinoline. Red-violet and garnet shades are obtained on wool by the single-bath chrome method. C. H.

**[Manufacture of tris]azo dyes.** IMPERIAL CHEM. INDUSTRIES, LTD., and C. PAINE (B.P. 397,879, 26.11.31).—A 1-*m*- or *p*-aminoaryl-3-methyl(or carboxy)-5-pyrazolone or its sulphonic acid is used as 2nd component in dyes  $A \rightarrow P \rightarrow M \rightarrow E$ , where  $A$  = an amine of the  $\text{C}_6\text{H}_6$  or  $\text{C}_{10}\text{H}_8$  series,  $M$  = a usual middle component, and  $E$  =  $\alpha$ - or  $\beta$ - $\text{C}_{10}\text{H}_7\cdot\text{OH}$ ,  $\alpha$ - or  $\beta$ - $\text{C}_{10}\text{H}_7\cdot\text{NH}_2$ , or an aminonaphthol, or a sulphonic acid of these, or a hydroxynaphthoic acid, or an *N*-substituted naphthylamine- or aminonaphthol-sulphonic acid,  $E$  being other than a 1:8-aminonaphtholsulphonic acid or *N*-acyl derivative thereof when  $M$  is a 1-amino- $\beta$ -naphthyl ether or derivative thereof. Where  $E$  contains in an *N*-substituent a diazotisable  $\text{NH}_2$ -group, the dyes may be developed on the fibre with the usual developers. Examples include: sulphanilic acid  $\rightarrow P \rightarrow$  2:5-dimethoxyaniline  $\rightarrow p$ -aminobenzoyl-S-acid (blue-green; developed with phenylmethylpyrazolone, yellow-green); sulphanilic acid  $\rightarrow P \rightarrow$  2-ethoxy-Cleve acid  $\rightarrow R$ -acid (blue-green); Brönner acid  $\rightarrow P \rightarrow$  Cleve acid  $\rightarrow o$ -chlorobenzoyl-H-acid (yellow-green). C. H.

**Indigoid and azo dyes.**—See III. **Photosensitising dyes.**—See XXI.

#### V.—FIBRES; TEXTILES; CELLULOSE; PAPER.

**Characteristics of the principal textile fibres and their detection.** T. PAVOLINI (L'Ind. Chimica, 1933, 7, 1245—1251).—A review.

**Manufacture of leather cloth.** A. JONES (Synth. and Appl. Finishes, 1933, 4, 208—211).—A brief description.

**Viscosity of cuprammonium solutions of cellulose.** A. I. GERSCHON (J. Appl. Chem. Russ., 1933, 6, 721—729).—A viscosimeter is described, in which the free space after introduction of cellulose solution (I) is filled with Hg, with which (I) is shaken, and the rate of flow is measured after settling of the Hg emulsion. R. T.

**Ethylation [of cellulose].** II. N. I. NIKITIN and T. I. RUDNEVA (J. Appl. Chem. Russ., 1933, 6, 716—720).—The facility of ethylation increases in the order  $\text{EtCl} < \text{EtBr} < \text{EtI}$ , a max. of 2.75 Et per  $\text{C}_6\text{H}_{10}\text{O}_5$  unit being obtained by heating EtI at  $120^\circ$  during 8 hr. with cellulose in 40% NaOH. The no. of  $\text{CH}_2\text{Ph}$  groups introduced into ethylcelluloses varies inversely with the no. of Et groups present, total substitution in no case affecting  $> 3$  OH groups. The product containing  $2\frac{3}{4}$  OEt and  $\frac{1}{2}$   $\text{O}\cdot\text{CH}_2\text{Ph}$  per  $\text{C}_6\text{H}_{10}\text{O}_5$  gives films possessing a high degree of transparency and elasticity. R. T.



**Lesser-known uses of viscose.** H. HOFFMANN (Papier-Fabr., 1933, 31, 497—500).—Viscose may be used as a sizing agent, but although it increases the strength of the paper, especially the wet strength, it produces a yellow discoloration and is thus used chiefly for wrappings. It also forms an ideal finishing agent for fabrics. Methods are described for producing from it artificial sausage skins and sponges. D. A. C.

**Benzylation of cellulose under technical conditions.** II. N. I. NIKITIN and M. A. AVIDON (J. Appl. Chem. Russ., 1933, 6, 710—715).—Cotton-wool (1 pt.) is soaked during 18—24 hr. in 25% NaOH, which is then squeezed out until the product has 3 times its original wt., when it is heated at 100° with 2.6 pts. of  $\text{CH}_3\text{PhCl}$  (I) during 3 hr., 2 pts. of NaOH are added, and the mixture is heated for a further 4 hr. The benzylcellulose obtained contains  $> 2 \text{ O-CH}_2\text{Ph}$  per  $\text{C}_6\text{H}_{10}\text{O}_5$  unit, has a relative viscosity of 2.3—4.8, and is 90—99% sol. in  $\text{C}_6\text{H}_6\text{-EtOH}$ . 34% of the NaOH introduced is used in the side-reaction of hydrolysis of (I). (I) remaining after completion of the reaction is recovered by extraction with benzene, and residual NaOH by extraction with  $\text{H}_2\text{O}$ . R. T.

**Technology of cellulose derivatives.** K. MIENES and G. VON FRANK (Angew. Chem., 1933, 46, 583—584).—The properties of cellulose acetate and nitrate and of ethyl- and benzyl-cellulose are summarised, and their use in the manufacture of lacquers, plastics, foils, and photographic films are indicated. A. G.

**Factors influencing chlorination of [unbleached] Mitscherlich [spruce] sulphite pulp.** O. KRESS and E. H. VOIGTMAN (Paper Trade J., 1933, 97; T.A.P.P.I. Sect., 73—88).—The concn. of the stock during chlorination with  $\text{Cl}_2$  gas has a marked effect on both the chemical and physical properties of the resulting pulp. The higher is the consistency, especially above 40%, the lower are the cuprammonium viscosity (I) and the  $\alpha$ -cellulose content (II), and the higher the bleachability (III) and the lignin content (IV). The resulting bleached pulps, however, unexpectedly possess identical (I), although their physical strength characteristics are lower in direct relation to (I) after the first stage. The time and rate of chlorination do not affect pulp quality, nor does the temp. of the  $\text{Cl}_2$ . The temp. of the stock, on the other hand, has an appreciable effect; the higher is the temp. the lower does (I) and the higher does (III) become. HCl under the range of concn. and temp. investigated does not degrade the pulp. Increasing amounts of  $\text{Cl}_2$  up to 50% of the max. consumption, followed by treatment with CaO, do not affect (I) or pentosan content, but reduce (III) and (IV) linearly, and (II) slightly; above 50% (I) falls very rapidly. The OMe content (V) also falls from 0.8 to 0.4% with increasing amounts of  $\text{Cl}_2$ , at which lower val. it remains const. regardless of (IV). This residual (V) suggests that some OMe groups are present which are not intimately combined with the lignin. Potentiometric titration data suggest that chlorination is almost entirely a substitution reaction, with the formation of HCl equiv. to half the Cl introduced. About 15% of the  $\text{Cl}_2$  combines with lignin to form products which in absence of CaO are insol.

in the acidic residue, but are sol. in  $\text{H}_2\text{O}$ . When causticised with CaO almost all the Cl is found in the liquor. Chlorination, in  $\text{CCl}_4$  or in  $\text{H}_2\text{O}$ , of lignins isolated by three different methods shows that the properties of the products depend on the solvent. Those from  $\text{CCl}_4$  are insol. in  $\text{H}_2\text{O}$  and in 5% HCl, and only slightly sol. in 5% KOH; those from  $\text{H}_2\text{O}$  are slightly sol. in  $\text{H}_2\text{O}$  and HCl but completely sol. in KOH. Furthermore, those from  $\text{CCl}_4$  contain almost twice as much Cl as those from  $\text{H}_2\text{O}$ . It is suggested that one or other solvent causes preferential chlorination of the side-chain of the lignin mol. The chlorolignins obtained when  $\text{H}_2\text{O}$  is the solvent behave very like the lignin in chlorinated pulp. H. A. H.

**Methods of determining the strength of wood cellulose.** Soc. SPIRO (Chim. et Ind., 1933, 29, Spec. No., 1101—1106).—Details are given of Swedish, American, and German indirect methods, using test sheets prepared in a standard fashion, and of a modified method in which a special form of apparatus (described) is employed. A. G.

**Permanganate-reducing power of paper half-stuff.** E. WATHELET and L. RAMCLE (Monit. Pap. belge, 1933, 13, 27—31; Chem. Zentr., 1933, i, 2485).—Reducing org. impurities present in wood cellulose are determined by making 2 g. of dried cellulose into a ball with 8 g. of  $\text{H}_2\text{O}$  and placing it for 1 hr. in a mixture of 22 c.c. of  $N\text{-H}_2\text{SO}_4$ , 50 c.c. of 0.1N- $\text{KMnO}_4$ , and 470 c.c. of  $\text{H}_2\text{O}$ , then adding 50 c.c. of 0.1N- $\text{FeSO}_4\cdot(\text{NH}_4)_2\text{SO}_4\cdot 6\text{H}_2\text{O}$ , filtering, diluting to 1 litre, and titrating with 0.1N- $\text{KMnO}_4$ . The  $\text{KMnO}_4$  val. is the vol. used for the whole filtrate  $\times 0.31382$ . Bleached material gives vals. of 0.09—0.85%, unbleached bleachable material 3.11—5.46%, unbleachable material 7.34—9.84%. A. A. E.

**Capillary rise of water in fibrous sheets and possible applications.** F. A. SIMMONDS (Paper Trade J., 1933, 97; T.A.P.P.I. Sect., 116—118).—There is a definite relationship between the height of the capillary rise of  $\text{H}_2\text{O}$  in strips of certain types of pulp and unsized paper sheets, which can be used for measuring the effective capillary diam. parallel to the upper and lower surfaces of the sheet, and thus affording a possible indication of the uniformity of structure.  $\text{H}_2\text{O}$  is unsuited for testing calendered and sized papers in this manner since the rate of absorption is too small. The possibility of utilising the swelling velocity coeff. of pulps as a means of predicting the rate of hydration during beating is indicated. H. A. H.

**Determination of zinc sulphide pigments in paper.** F. A. STEELE (Paper Trade J., 1933, 97; T.A.P.P.I. Sect., 103—104).—Details of a method [using  $\text{K}_4\text{Fe}(\text{CN})_6$  with  $\text{NHPh}_2$  as indicator] of determining  $\text{ZnS}$ , in absence and in presence of other paper fillers, are given. When Ba is also present (as when lithopone is the filler) a separate analysis must be made. H. A. H.

**Methods of testing transparent papers.** ANON. (Brit. Plastics, 1933, 5, 114, 125, 146—148).—Simple chemical methods of differentiating between regenerated cellulose, cellulose acetate, and gelatin films are described. Their origins of manufacture may be detected by



examining their surface characteristics by means of shadow pictures. D. A. C.

**Faults in printing paper and their detection.** H. VNUK (Chim. et Ind., 1933, 29, Spec. No., 1094—1100).—Methods are given for the examination of stains. Yellowing when exposed to light is due to lignin from wood pulp, to Fe, or to resin. Other faults are unequal wt. of sheets, tendency to crinkle, roll, or dust, and large changes of dimensions with variations of atm. humidity. A. G.

**Filter-press cloth.**—See I. **Dry distillation of waste sulphite liquor.** Paraffin wax sizes.—See II. **Testing welds in sulphite liquor.**—See X. **Cellulose esters for cable insulation.**—See XI. **Filastic.**—See XIV. **Tanning with sulphite-cellulose waste extract.**—See XV. **EtOH from wood.** **Fermentation of cellulosic materials.**—See XVIII.

See also A., Oct., 1009, **Limits of fractionation of cellulose nitrates.** 1027, **Collodion thimbles for ultra-filtration.** 1038, **Additive compounds of cellulose.** **Nitration of cellulose.** **Introduction of  $\text{NH}_2$ -groups into cotton cellulose.** 1050, **KOH fusion of jute-lignin.** 1051, **Oxidation of alkali-lignin.** **Lignin and its derivatives.** 1083, **Fermentation of cellulose.**

#### PATENTS.

**Treatment of fibrous plants for recovery of the fibres thereof.** B. B. WOOD (B.P. 398,033, 22.9.32. N. Zealand, 12.10.31).—An explosion process is described in which the plant material (sisal, flax, or jute), preferably after soaking in  $\text{H}_2\text{O}$  under vac., is subjected to heat and pressure in a closed chamber from which it is subsequently explosively discharged into an evacuated vessel. Suitable apparatus is described. D. J. N.

**Production of fibre.** S. D. WELLS, ASSR. to MINE & SMELTER SUPPLY CO. (U.S.P. 1,894,577, 17.1.33. Appl., 17.9.27).—The best results are obtained in the rod mill when the consistency of the pulp is 5–9.5 pts. per 100 pts. of  $\text{H}_2\text{O}$ . D. J. N.

**Manufacture and treatment of filaments and the like.** H. DREYFUS (B.P. 397,881, 29.1.32).—The stretching of cellulose acetate (I) (etc.) filaments, continuously with or subsequent to their production, by applying solvents or swelling agents to the filament before or during stretching is facilitated if there is added to the spinning solution 5–15% [on the wt. of (I)] of a substance (liquid or solid) which remains in the filament during spinning and is subsequently removed (by dissolution) before or during application of the stretch-assisting agent. Suitable substances include glycerin, cyclohexanol and its derivatives, oils, fats, waxes, and the usual plasticisers. D. J. N.

**Dehydration of nitrocellulose.** A. LANGMEIER, ASSR. to HERCULES POWDER CO. (U.S.P. 1,901,561, 14.3.33. Appl., 24.5.27).—After removal of excess  $\text{H}_2\text{O}$  by mechanical means, amyl alcohol (A) is caused to percolate through the damp nitrocellulose, the product being pressed or centrifuged until the A content is 20–40%. W. J. W.

**Manufacture of wood pulp.** M. C. TAYLOR and J. F. WHITE, ASSRS. to MATHIESON ALKALI WORKS, INC.

(U.S.P. 1,894,501, 17.1.33. Appl., 17.4.30. Renewed 19.5.32).—An alkali or alkaline-earth chlorite is used as the digesting agent at temp. up to  $100^\circ$  or more (according to the pressure), preferably in neutral or alkaline solution. Thus poplar chips are digested at  $100^\circ$  for about 16 hr. with CaO and  $\text{Ca}(\text{ClO}_2)_2$  (I) in  $\text{H}_2\text{O}$ , a further quantity of (I) being added towards the end of the cook. The pulp may be further bleached by washing with dil. HCl (0.4 g./litre) or aq.  $\text{NaHSO}_3$ . The cellulose is not destructively attacked by the treatment. D. J. N.

**Producing a white filler product from sulphate-pulp lime mud.** O. KRESS (U.S.P. 1,894,620, 17.1.33. Appl., 26.6.29).—The lime mud is treated with an oxidising agent (5–10% of  $\text{CaOCl}_2$  on dry solids) to oxidise FeS etc. and is then washed. D. J. N.

**Heat recovery [from hot pulp liquors].** D. K. DEAN, ASSR. to FOSTER WHEELER CORP. (U.S.P. 1,894,758, 17.1.33. Appl., 1.11.28).—Heat from digester discharges is recovered by flashing the liquor (A) into steam in two stages, each comprising an evaporator and condenser, A in passing from the first to the second stage forming a column affording the necessary difference in pressure. B. M. V.

**Stencil paper.** K. W. CARR, ASSR. to DITTO, INC. (U.S.P. 1,894,731, 17.1.33. Appl., 29.9.26).—Yoshino paper is coated with a solution (in org. solvents or solvent mixtures) of a heat-hardening synthetic resin, preferably a phenol-tung oil- $\text{CH}_2\text{O}$  resin, containing shortening agents (Al stearate,  $\text{BaSO}_4$ , or waxes) and a softening agent (neatsfoot oil + Bu phthalate), and is then heated to, e.g.,  $140^\circ$  to polymerise the resin. D. J. N.

**Manufacture of parchment-like paper.** ALADDIN INDUSTRIES, LTD., ASSEES. OF F. O. REISS (B.P. 397,550, 26.2.32. U.S., 27.2.31).—Unsize paper made from high- $\alpha$ -cellulose sulphite pulp is impregnated (at  $140$ – $160^\circ$  for 1–3 min.) with a molten, partly polymerised glyptal resin plasticised with, e.g.,  $(\text{C}_6\text{H}_4\text{Me})_3\text{PO}_4$ , and the resin is then completely polymerised, e.g., at  $130^\circ$  for 6 hr. D. J. N.

**Paper manufacture.** J. H. SWAN, 3rd, ASSR. to GARDNER-RICHARDSON CO. (U.S.P. 1,895,527, 31.1.33. Appl., 30.8.29).—Multi-ply box board with hard sized liners, and preferably containing an inner layer of asphalt, is coated on one side first with a solution containing, e.g., 75 vols. of 38% aq. Na silicate, 25 vols. of glycerin, and 1–2% of alkali ( $\text{Na}_2\text{CO}_3$ ), and then, after drying, with (paraffin) wax. Such a board is moisture- and grease-proof and suitable for foodstuffs. D. J. N.

**Manufacture of indurated cellulosic product.** D. W. HOLLINGWORTH and L. W. TARR, ASSRS. to CONTINENTAL-DIAMOND FIBRE CO. (U.S.P. 1,894,907, 17.1.33. Appl., 8.1.32).—Unsize paper (about 0.01 in. thick and containing, e.g., 2%  $\text{H}_2\text{O}$ ), preferably made from cotton or high- $\alpha$ -cellulose pulp, is treated with a  $\text{H}_2\text{SO}_4$ - $\text{AcOH}$ - $\text{H}_3\text{PO}_4$  mixture containing < 25 wt.-%  $\text{H}_2\text{O}$  at room temp. for a few sec. and is then built up to give a product of the required thickness. This is allowed to "set," e.g., for 2 hr. in air of 25–40% R.H. at  $15$ – $21^\circ$ , and is then washed and dried. The product has the desirable properties of vulcanised fibre, but is superior thereto. D. J. N.



**Determining H<sub>2</sub>O in textiles. Filter diaphragms etc.**—See I. **AcOH from cellulose derivatives.**—See III. **Strengthened glass.**—See VIII. **Ink for cheque protection.**—See XIII. **Distillable spirits by fermentation.**—See XVIII. **Nitrocellulose powders.**—See XXII.

## VI.—BLEACHING; DYEING; PRINTING; FINISHING.

**Scouring of linen.** E. BUTTERWORTH and H. A. ELKIN (J. Text. Inst., 1933, 24, p 217—225).—If linen is scoured with a series of NaOH solutions of concns. 0–2.5% the loss of wt. increases, and the Cu no. decreases, with increasing concn. The viscosity of a filtered cuprammonium solution remains const. Except at high concns., the consumption of NaOH, as determined by conductivity titration, is proportional to the loss of wt. There is a loss of total alkali, which is ascribed to sorption. A. G.

**[Textile] fulling and milling machinery.** E. K. SCOTT (J. Text. Inst., 1933, 24, p 247—263).—Various types of fulling stocks and of rotary milling machines are described. A. G.

**Performance of two new finishing machines on linen.** E. BUTTERWORTH (J. Text. Inst., 1933, 24, p 209—216).—A new 3-bowl, V-type friction calender for refinishing single cloths and an all-metal beetling engine are described. A. G.

**Modern [textile] finishing machinery.** W. J. NORTON (J. Text. Inst., 1933, 24, p 231—246).—Machines are described for bleaching in open width, for cloth-mercerising without a stenter, for jigger-dyeing at const. cloth speed and min. tension, for multiple chasing at full pressure, and for sanforising; improved stenter clips and driving mechanism for light cloths are also described. A. G.

**Recent developments in textile finishing.** L. G. LAWRIE (J. Text. Inst., 1933, 24, p 226—230).—A short description is given of sulphonated and sulphated fatty alcoholic detergents, of wetting and emulsifying agents, of immunised cotton, of the anti-crease process, and of new fixing and stripping agents for dyes. A. G.

**Viscose [as finishing agent].**—See V. **Wetting-out agents etc.**—See XII. **Filastic.**—See XIV. **Tanning fishing nets.**—See XV.

## PATENTS.

**Dyeing.** IMPERIAL CHEM. INDUSTRIES, LTD., C. H. BARLOW, and L. G. LAWRIE (B.P. 398,175, 31.12.31).—Cellulose, regenerated cellulose, wool, and/or silk materials are treated, before or after dyeing, with a dil. aq. solution of a heterocyclic NH<sub>4</sub> salt carrying on the ring-N an alkyl > C<sub>11</sub>, acyl, hydroxyalkyl, and hydroxyalkylcyl groups being absent. C. H.

**Dyeing or printing of textile materials consisting of or containing cellulose esters and ethers.** SOC. CHEM. IND. IN BASLE (B.P. 396,893, 19.5.32. Switz., 23.5.31. Acetate silk etc. is dyed with a condensation product of an aromatic amino-aldehyde or nitrosoarylamine (the NH<sub>2</sub> may carry hydrocarbon substituents) with a compound containing a reactive Me or CH<sub>2</sub>

group. Examples include products from: *p*-dimethylaminobenzaldehyde with 1-*o*-chlorophenyl-3-methyl-5-pyrazolone (red-yellow), CN·CH<sub>2</sub>·CO<sub>2</sub>Et (green-yellow), *p*-dimethylaminostyryl Me ketone (orange), barbituric acid (pink), or 5-chloro-2-methylindole (violet); nitroso-dimethylananiline with 1-phenyl-3-methyl-5-pyrazolone (violet-bordeaux), CH<sub>2</sub>(CN)<sub>2</sub> (orange), or thioindoxyl (violet-red). C. H.

**Production of coloured cellulose derivatives.** R. DINKLAGE (B.P. 398,279, 12.4.32. U.S., 18.4.31).—Chromophore and auxochrome groups are introduced into an aromatic residue combined with the cellulose mol. Examples are: cellulose condensed with PhNO<sub>2</sub> in conc. H<sub>2</sub>SO<sub>4</sub>, reduced, → β-C<sub>10</sub>H<sub>7</sub>·OH; with *p*-NO<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·CO·CO·CH<sub>2</sub>Cl, condensed with (CH<sub>2</sub>Ph)<sub>2</sub>CO to the cyclopentadienone, reduced, → Naphthol AS; with *p*-NO<sub>2</sub>·C<sub>6</sub>H<sub>4</sub>·CH<sub>2</sub>Cl, reduced, → Naphthol AS; with Mg *p*-nitrobenzoate, *p*-nitrobenzoylated or acetylated, reduced, → Naphthol AS. C. H.

**Fast dyeing with direct dyes.** BLEACHERS' ASSOC., LTD., C. L. WALL, and F. FARRINGTON (B.P. 398,532, 14.3.32).—A direct aminoazo dye is diazotised on the fibre and coupled with a 2:3-hydroxynaphthoic arylamide or acetoacetic tolidide etc. Suitable aminoazo dyes are Diazo-fast-yellow 2G, Diazo-sky-blue 3GL, Diazo-brilliant-green 3G, etc. C. H.

**[Production of] fast tints on wool [ice colours].** A. G. BLOXAM. From SOC. CHEM. IND. IN BASLE (B.P. 396,859, 31.3.32).—A 2:3-hydroxynaphthoic arylamide free from SO<sub>3</sub>H and SO<sub>2</sub>·NHR groups is coupled on wool with a diazobenzene carrying in position 3 or 4 an acylamino-group and in positions 4:6 or 3:6 H, halogen, alkyl, or alkoxy. For 1 pt. of wool, 6–50 pts. of grounding liquor (I) must be used, containing > 0.05 pt. of arylamide and 0.075–0.125 pt. of NaOH [which must be > 4 g. per litre of (I)]; the grounding must practically exhaust the bath. Examples are: 4-benzamido-2:5-dimethoxyaniline → *p*-chloroanilide (red-blue); 6-benzamidocresidine → anilide (violet). C. H.

**Manufacture of water-insoluble azo dyes on the fibre [ice colours].** I. G. FARBEIND. A.-G. (B.P. 397,016, 18.1.33. Ger., 18.1.32).—Diazo or tetrazo compounds free from solubilising groups are coupled on the fibre with a 2:3-hydroxynaphthoic 6-alkoxy-β-naphthylamide to give light-fast dyeings. Examples are the MeO- and EtO-compounds with diazotised *m*-4-xyldine (red), 2:3-dichloro-*p*-toluidine (blue-red), 5-nitro-*o*-anisidine (bordeaux), 2:4:5-trichloroaniline (orange-brown), 4-benzamido-2:5-dimethoxyaniline (reddish-dark blue), or *o*-anisidine → α-naphthylamine (black). C. H.

**Dyeing [with ice colours].** IMPERIAL CHEM. INDUSTRIES, LTD., M. MENDOZA, and W. A. SEXTON (B.P. 398,163, 29.1.32).—Claret shades fast to kier-boiling and Cl<sub>2</sub> are obtained by coupling a 2:3-hydroxynaphthoic arylamide with the tetrazo compound of a diamine prepared by condensing 2 mols. of an *o*-alkoxyaniline and 1 mol. of an aromatic aldehyde, all the components being void of solubilising and NO<sub>2</sub>-groups. Examples of tetrazo components are products from: cresidine with PhCHO (m.p. 183–184°) or *o*-C<sub>6</sub>H<sub>4</sub>Cl·CHO;



*o*-anisidine with *m*-methoxybenzaldehyde (m.p. 85–90°) or PhCHO. C. H.

Wetting etc. agents. Detergents.—See III.

## VII.—ACIDS; ALKALIS; SALTS; NON-METALLIC ELEMENTS.

(A) Vanadium, (B) manganese, catalysts in sulphuric acid manufacture. M. O. CHARMANDARIAN and K. I. BRODOVITSCH (Ukrain. Chem. J., 1933, 8, 49–57, 58–65).—(A) The activity of  $V_2O_5$ -zeolite catalyst in oxidation of  $SO_2$  is augmented by addition of other oxides: thus, with  $Al_2O_3$  33,  $Fe_2O_3$  35,  $CoO$  47,  $NiO$  51,  $UO_3$  91, and  $PbO$  99% conversion into  $SO_3$  takes place at 450°. The  $PbO$ - $V_2O_5$ -zeolite catalyst is unstable, % conversion falling to 79 after 6 days. A stable catalyst, unaffected by  $H_2O$  vapour, is prepared by mixing aq.  $Na_2SiO_3$ ,  $Pb(OAc)_2$ ,  $NH_4VO_3$ ,  $H_2SO_4$ , and alum, and drying at 100°. Greater stability and activity are obtained by exposing the freshly-prepared catalyst to gradually increasing concns. of  $SO_2$ . (B) The % conversion of  $SO_2$  into  $SO_3$  by a no. of catalysts prepared by adding salts to aq.  $Na_2SiO_3$  increases in the series:  $Al_2(SO_4)_3 < Fe_2(SO_4)_3 < MnCl_2 \cdot NiCl_2$  (3:1)  $< CoCl_2 < NiCl_2 < MnSO_4 < MnSO_4 \cdot Pb(NO_3)_2$  (3:1)  $< MnSO_4 \cdot Pb(NO_3)_2$  (1:1)  $< MnSO_4 \cdot Pb(NO_3)_2$  (9:1)  $< MnCl_2 \cdot CoCl_2$  (3:1)  $< MnCl_2 < MnCl_2 \cdot Pb(OAc)_2$  (3:1)  $< MnCl_2 \cdot Fe_2(SO_4)_3$  (3:1)  $< MnCl_2 \cdot CuCl_2$  (3:1). Max. conversion attains 96% at 450°, but the activity of the catalysts falls rapidly with time, probably owing to occupation of the active centres by  $SO_3$ . Alum does not stabilise the catalysts. R. T.

Application of conductivity measurement of nitric acid concentration to plant control. H. M. COSTER (Ind. Eng. Chem., 1933, 25, 980–982).—Electrical conductivity (*C*) curves for pure  $HNO_3$  diluted with varying proportions of  $H_2O$  show some variation from those obtained with acid made from  $NaNO_3$ , principally because the presence of  $N_2O_4$  increases *C* in acid of 90–100% concn. In the case considered, where it was desired to make a cut when the acid distilled fell below the given concn., this point did not matter. A continuous sample was taken and *C* compared with 70%  $HNO_3$  on a bridge, Pt electrodes being used. A by-pass enabled pressure and temp. to be controlled, and the method is instantaneous. C. I.

Utilisation of dilute hydrochloric acid obtained in the production of sodium silicofluoride. V. J. ANOSOV and V. F. UST-KATSKHINTZEV (J. Appl. Chem. Russ., 1933, 6, 630–632).—The solution remaining after pptn. of  $Na_2SiF_6$  (a by-product of the superphosphate industry), containing  $HCl$  1.35,  $NaCl$  1.65,  $SiF_6$  0.67, and  $SO_4$  0.03%, can be used, after addition of sufficient  $H_2SO_4$  to raise its  $HCl$  content to 2.6%, for extraction of phosphorites, when about 80% extraction is obtained. The extract contains 1–1.4%  $P_2O_5$ , which is pptd. as  $Ca_3(PO_4)_2$ . R. T.

Manufacture, handling, and use of hydrocyanic acid. P. J. CARLISLE (Ind. Eng. Chem., 1933, 25, 959–964).—Commercial liquid HCN is a 97% aq. solution having  $d_{25}^{25}$  0.692. It is stabilised by a trace of  $H_2SO_4$ ; decomp. is promoted by  $H_2O$  and alkalis, the

first sign being development of a yellow colour. A 23% NaCN solution is mixed continuously with a slight excess of  $H_2SO_4$  in a closed apparatus with a special device to effect quick mixing. About 80% of the HCN is liberated as gas, and the remainder is recovered with steam in another vessel. The vapours pass through a cooler to a still with reflux condenser. From this the exit vapour is maintained at 32° and on condensation yields 97% HCN solution.  $CO_2$  derived from  $Na_2CO_3$  in the NaCN is finally removed by warming. Antimonial Pb is used for apparatus in contact with  $H_2SO_4$  and thenceforward Al or monel metal. The acid is sent out in 100-lb. drums (ice-cooled during transport). For fumigation, trees are covered with canvas tents and a measured dose is sprayed underneath, liquid acid being kept away from the leaves. C. I.

Conductometric methods for determination of alkalis in lime and lime-water. K. ŠANDERA (Z. Zuckerind. Czechoslov., 1933, 58, 17–19).—The filtrate from an aq. suspension of  $Ca(OH)_2$  is neutralised to phenolphthalein with  $H_2C_2O_4$  and the electrical conductivity is determined. A method for the direct calculation of alkali content is worked out. E. S. H.

New processes for manufacture of ammonium nitrate. L. TAMISÉ (Chim. et Ind., 1933, 29, Spec. No., 906–920).—Economies are effected by using the heat of neutralisation of the  $HNO_3$  and  $NH_3$  for the concn. of the  $NH_4NO_3$  solutions in stages of increasing vac., the steam being used for preheating the acid. Crystallisation is carried out in vac., whereby use is made of the heat of crystallisation for drying; the product contains < 0.5%  $H_2O$ . A. A. L.

Reduction of sodium sulphate in briquettes. G. J. TARASOV and A. S. IZOTOVA (J. Appl. Chem. Russ., 1933, 6, 633–637).—92–93% yields of  $Na_2S$  are obtained by heating briquettes containing 83.4%  $Na_2SO_4$  and 16.6% C during 20 min. at 850°. Apparatus is described for this process on a factory scale. R. T.

Preparation of sodium ferrite by Löwig's process. I. M. O. CHARMANDARIAN and G. V. MARTSCHENKO (Ukrain. Chem. J., 1933, 8, 95–103).—Na ferrite is prepared at 850° from 3:1 mixtures of finely-powdered Fe ore and  $Na_2CO_3$ . R. T.

Extraction of potash [potassium sulphate] from polyhalite. III. Effect of particle size, sodium chloride concentration, and temperature on hot extraction by a multistage process. J. E. CONLEY and F. FRAAS (Ind. Eng. Chem., 1933, 25, 1002–1009).—The extraction of  $H_2SO_4$  (I) from calcined polyhalite with  $H_2O$  was studied, using a no. of batches in order to simulate large-scale countercurrent working. Relatively coarse material (10-mesh) was found quite suitable. The presence or addition of up to 20% of NaCl on the wt. of (I) considerably improves extraction, and in this case a solution of 11 g. of (I) per 100 c.c. of  $H_2O$ , with 97–98% efficiency, should be obtained. The temp. of extraction should be 100° except in the final stage. C. I.

Manufacture of potassium salts from polyhalite and their significance as fertilisers. E. P.



PARTRIDGE and A. H. EMERY (Amer. Fertiliser, 1933, 79, No. 6, 5—8; No. 7, 7—8).—A review. A. G. P.

**Velocity of calcination of chalk.** K. SMOLEŃSKI and H. IWANIK (Prace Centr. Lab. Cukrownicz., 1928—1931, 145—159; Chem. Zentr., 1933, i, 2453).—A study of marble and 15 other varieties of  $\text{CaCO}_3$  is reported. A. A. E.

**Use of starch for accelerating velocity of settling of sludge.** A. V. PETROV (Ukrain. Chem. J., 1933, 8, 89—94).—The rate of settling of  $\text{CaCO}_3$  suspended in 10%  $\text{NaOH}$  is 5 times as great in presence of 0.01% starch as in its absence. R. T.

**Modern bleaching powder manufacture.** ANON. (Ind. Chem., 1933, 9, 300—304).—The Backman plant at Staveley is described and illustrated. D. K. M.

**Preparation of calcium chromate in the wet way.** J. MILBAUER and J. DOŠKAŘ (Chim. et Ind., 1933, 29, Spec. No., 784—786).— $\text{CaCrO}_4$  is obtained in best yield by adding to a cold saturated solution of  $\text{Na}_2\text{CrO}_4$  a solution containing 400—450 g. of anhyd.  $\text{CaCl}_2$  per litre of  $\text{H}_2\text{O}$ , using a 16% excess. With such conc. solutions, and still more with heating, considerable occlusion of  $\text{CaCl}_2$  and  $\text{NaCl}$  occurs. It is inadvisable to use  $\text{CaO}$ . C. I.

**Decomposition of raw phosphates by nitric acid.** A. GUYER and A. LIKIERNIK (Helv. Chim. Acta, 1933, 16, 1033—1044).—The action of  $\text{HNO}_3$  on crude (mineral)  $\text{Ca}_3(\text{PO}_4)_2$  has been examined with respect to the relative amounts of  $\text{H}_2\text{O}$ -sol., citrate-sol., and insol.  $\text{P}_2\text{O}_5$  formed, acidity of the aq. extract, loss of N during the reaction, and loss of N on drying. The results are given in curves and tables. F. L. U.

**Preparation of barium chloride: I. From chlorine and barium sulphide. II. From chlorine and barium sulphate. III. From hydrogen chloride and barium sulphate.** M. O. CHARMANDARIAN and K. I. BRODOVITSCH (Ukrain. Chem. J., 1933, 8, 110—115, 116—118, 119—124).—I. Unsatisfactory yields of  $\text{BaCl}_2$  are obtained by passing  $\text{Cl}_2$  through aq.  $\text{BaS}$  suspensions at 85—95°, 10% yields are obtained by passing dry  $\text{Cl}_2$  over  $\text{BaS}$  at 150°, and 50% yields by mixing the  $\text{Cl}_2$  with  $\text{HCl}$  or  $\text{H}_2\text{O}$ ; at 200° 80—90% yields are obtained using  $\text{Cl}_2$  alone or together with  $\text{HCl}$ .

II. Up to 99% yields of  $\text{BaCl}_2$  are obtained by passing  $\text{Cl}_2$  through a 1:1 mixture of  $\text{BaSO}_4$  and C containing  $\text{Al}_2\text{O}_3$  catalyst, at 600°.

III. 96% yields of  $\text{BaCl}_2$  are obtained by passing  $\text{HCl}$  through  $\text{BaSO}_4$  at 800° during 3 hr., or at 1000° during 1 hr.; addition of  $\text{FeCl}_3$  or  $\text{SiO}_2$  gel retards reaction, whilst addition of an equal wt. of C to the  $\text{BaSO}_4$  gives 94% conversion after 3 hr. at 600°. R. T.

**Manufacture of aluminium sulphate at the Dalecarlia Filter Plant, Washington, D.C.** C. J. LAUTER (Ind. Eng. Chem., 1933, 25, 953—958).—Bauxite is ground (to pass 60-mesh) and conveyed pneumatically to storage bins. The ore should contain 57%  $\text{Al}_2\text{O}_3$  and  $> 1.5\%$   $\text{H}_2\text{O}$  at 100° and give  $< 29\%$  loss on ignition. It is mixed with  $\text{H}_2\text{SO}_4$  (d 1.70) in boiling-tanks (T) of Pb-lined steel holding 12 tons,  $\text{H}_2\text{O}$  being added as necessary to control the reaction. It is then diluted, set aside for 12 hr., stirred with air, and adjusted for  $p_{\text{H}}$  by  $\text{Ba}(\text{OH})_2$  titration. The solution

is used direct for  $\text{H}_2\text{O}$  treatment. The Pb lining of the T is in horizontal annular sections turned over steel supporting rings; pipe-lines are of Pb. Costs are discussed. C. I.

**Activation of earths.** A. TRAVERS (Chim. et Ind., 1933, 29, Spec. No., 793—796).—The activation of earths such as bentonite by treatment with 15%  $\text{H}_2\text{SO}_4$  or  $\text{HCl}$  is essentially due to the liberation of  $\text{SiO}_2$  as gel, and the optimum temp. of dehydration is the same as for  $\text{SiO}_2$  gel. Kaolin and the like are not attacked by dil. acids, and activation by hot conc.  $\text{H}_2\text{SO}_4$  though possible is costly. They may be converted into zeolites by fusion with  $\text{Na}_2\text{CO}_3$  or autoclaving with 10%  $\text{NaOH}$  and are then activated by dil. acids. The adsorptive power of the products is, however, not high. C. I.

**Detection and determination of nickel in commercial cobalt salts by means of formaldoxime.** G. DENIGÈS (Bull. Trav. Soc. Pharm. Bordeaux, 1932, 70, 106—107; Chem. Zentr., 1933, i, 2146).—Ni (0.1%) is determined by colorimetric comparison of solutions of Co salts (0.05 g.  $\text{Co}^{++}$  per litre) to which are added formaldoxime and  $\text{NaOH}$ . A. A. E.

**Determination of lead peroxide.** V. A. V. PAMFILOV and E. G. IVANČEVA (J. Gen. Chem. Russ., 1933, 3, 262—268; cf. B., 1932, 546).—The  $\text{H}_2\text{O}_2$  method gives consistent results and is to be preferred. R. T.

**Black mercury sulphide.** R. HUERRE (J. Pharm. Chim., 1933, [viii], 18, 145—153).—Commercial black  $\text{HgS}$  contains a large amount of free S (30—40%). A method for the determination of free insol. S is given. Insol. S combines more readily with Hg than sol. S. For the prep. of the black sulphide it is only necessary to use equiv. amounts of S and Hg. E. H. S.

**Radium industry of Portugal.** C. LEPIERRE and A. P. LEITE (Chim. et Ind., 1933, 29, Spec. No., 797—804).—About 10 g. of Ra were produced in Portugal during 1908—1926. The source is mixed sulphide ores in granite containing 0.5—1.5%  $\text{U}_3\text{O}_8$  and having the usual Ra-U ratio of  $3.2 \times 10^{-7}$ . Near the surface the ore is considerably sulphated. Both alkaline and acid treatment were necessary for extraction. The finely-ground ore was mixed with dil.  $\text{H}_2\text{SO}_4$ , a little  $\text{BaCl}_2$  added, and the solution filtered. The residue was autoclaved with conc.  $\text{Na}_2\text{CO}_3$ , filtered, and the carbonates were dissolved in  $\text{HCl}$  and pptd. with alternate additions of  $\text{BaCl}_2$  and  $\text{H}_2\text{SO}_4$  to carry down the Ra. Established practice is then followed. Costs are discussed. C. I.

**Polarographic determination of oxygen contained in industrial gases and waters.** V. VÍTEK (Chim. et Ind., 1933, 29, Spec. No., 215—217).—If the  $\text{O}_2$  in a gas is to be determined, a saturated solution of the gas is prepared and the polarographic record of the solution made.  $\text{O}_2$  concns. of 0.04—40 mg. per litre of solution can be determined in this way, as well as gaseous concns. of 100—0.5%. The method has been applied to mixtures of  $\text{O}_2$  with  $\text{H}_2$ ,  $\text{N}_2$ , air,  $\text{CO}_2$ , hydrocarbons, CO, rare gases, and furnace gases. Concns. of 0.04—0.01 mg. per litre may be determined approx. from the magnitude of the galvanometer oscillations when the Hg drops fall into the solution. J. W. S.



**Dry process for recovery of sulphur from hydrogen sulphide.** M. O. CHARMANDARIAN, E. L. KOPELIOVITSCH, and A. E. BURJAK (Ukrain. Chem. J., 1933, 8, 72—88).—Oxidation of  $\text{H}_2\text{S}$  with liberation of S (75—95% yields) takes place on passing the gas with a limited  $\text{O}_2$  supply over  $\text{SiO}_2$  gel,  $\text{Fe}_2\text{O}_3$ -clay, or  $\text{MnO}$ -clay catalyst at  $300^\circ$ . The heat of reaction is sufficient to maintain the temp. at  $300^\circ$  when the gas contains 8—10%  $\text{H}_2\text{S}$ . R. T.

**Catalytic production of chlorine from hydrogen chloride.** M. O. CHARMANDARIAN and G. V. PRICHODKO (Ukrain. Chem. J., 1933, 8, 68—71).—A catalyst consisting of  $\text{CuO}$ ,  $\text{Co}_2\text{O}_3$ ,  $\text{MnO}$ , and  $\text{Bi}_2\text{O}_3$  gives 97·5% yields of  $\text{Cl}_2$  from  $\text{HCl}$  at  $370^\circ$ . The catalyst is inactivated by  $\text{CO}$  or  $\text{SO}_2$ . R. T.

**S from city gas.**—See II. Metals in acid manufacture.—See X. Zn white.  $\text{Cr}_2\text{O}_3$  green. Chrome-yellow. By-products from lithopone prep. Analysing red lead.—See XIII. Rapid analysis of crude potash.—See XVII. Ageing of  $\text{Al}(\text{OAc})_3$  solutions.—See XX. Determining  $\text{CO}_2$  in air.—See XXIII.

See also A., Oct., 1011, Prep. of Zr sulphosalicylic acid jellies. 1013,  $\text{NaOH}$ - $\text{NaCN}$  melts. 1014, Causticisation of  $\text{Na}_2\text{CO}_3$  by  $\text{Fe}_2\text{O}_3$ . 1021, Prep. of  $\text{TiOH}$ , and of Ra-E. 1022, Determination of  $\text{H}_2\text{O}_2$ . 1022, Electrode potential of  $\text{Cl}_2$ .

#### PATENTS.

**Purification of battery [sulphuric] acid.** C. C. ROSE, Assr. to WILLARD STORAGE BATTERY CO. (U.S.P. 1,898,688, 21.2.33. Appl., 16.9.31).—Waste  $\text{H}_2\text{SO}_4$  containing  $\text{AcOH}$  and other wood acids derived from the original charging of accumulators containing wood separators is passed through a column of activated charcoal (I) to absorb the impurities. (I) is regenerated by blowing steam through it. A. R. P.

**Concentration of weak phosphoric acid.** C. B. CLARK, Assr. to AMER. CYANAMID CO. (U.S.P. 1,889,949, 6.12.32. Appl., 11.2.30).—Dil.  $\text{H}_3\text{PO}_4$  from the decomp. of phosphate rock (I) with  $\text{H}_2\text{SO}_4$  is used for scrubbing the hot gases containing  $\text{P}_2\text{O}_5$  and  $\text{CO}$  produced by electrothermal reduction of (I), whereby a more conc.  $\text{H}_3\text{PO}_4$  for the manufacture of superphosphate and a mist of  $\text{H}_3\text{PO}_4$ , which is condensed in an electrical precipitator, are obtained. A. R. P.

**Production of ammonia [from cyanamide].** P. H. FALTER, Assr. to AMER. CYANAMID CO. (U.S.P. 1,889,956, 6.12.32. Appl., 27.2.29).—Crude  $\text{CaCN}_2$  is added to spent liquor from a previous operation, and when the  $\text{C}_2\text{H}_2$  produced by the  $\text{CaC}_2$  present has escaped the mixture is heated in an autoclave with a small amount of  $\text{NaOH}$  or  $\text{Na}_2\text{CO}_3$  to  $> 160^\circ$ . When the pressure reaches 4 atm. the heat is cut off and the reaction allowed to proceed exothermically until the pressure reaches 16 atm. The  $\text{NH}_3$  is then allowed to escape into a condenser and absorption tower, the  $\text{CaCO}_3$  removed by filtration, and the liquor used again in the process. A. R. P.

**Production of phosphates.** C. (BARON) VON GIRSEWALD, H. WEIDMANN, and G. ROESNER, Assrs. to AMER. LURGI CORP. (U.S.P. 1,890,204, 6.12.32. Appl., 9.5.31. Ger., 19.5.30).—The Si content of ferrophosphorus is

reduced to  $< 5\%$  by fusion with  $\text{Fe}_2\text{O}_3$  or  $\text{Ca}_3\text{P}_2\text{O}_8$ , and the purified alloy is fused with  $\text{Na}_2\text{SO}_4$  to produce  $\text{Na}_3\text{PO}_4$  and  $\text{FeS}$ . A. R. P.

**Manufacture of [organic] dithiophosphates.** G. BARSKY and R. V. HEUSER, Assrs. to AMER. CYANAMID CO. (U.S.P. 1,889,943, 6.12.32. Appl., 8.8.29).—A solution of an aryl or alkyl alcohol in  $\text{C}_6\text{H}_6$ ,  $\text{PhMe}$ , or  $\text{CCl}_4$ , or mixtures of any of these, is treated with  $\text{P}_2\text{S}_5$  and, when the exothermic reaction ceases, the solution is filtered and treated with  $\text{NH}_2\text{Ph}$  to ppt. crystals of the  $\text{NH}_2\text{Ph}$  salt of the dithiophosphate, the solvent being used again in the process. A. R. P.

**Manufacture of tertiary organic phosphates.** W. W. GROVES. From CHEM. FABR. VON HEYDEN A.-G. (B.P. 398,659, 21.10.32).—The corresponding phosphite is oxidised, e.g., with  $\text{As}_2\text{O}_5$ ,  $\text{SO}_3$ ,  $\text{V}_2\text{O}_5$ , etc.;  $\text{Ph}_3\text{PO}$ , m.p.  $47\cdot8$ — $47\cdot9^\circ$ ,  $\text{Bu}_3\text{PO}$ , b.p.  $115$ — $118^\circ/1$  mm.,  $\text{Ph}_3(\text{o-C}_6\text{H}_4\text{Me})\text{PO}$ , b.p.  $255$ — $265^\circ/12$  mm., and  $\text{PhBu}_2\text{PO}$ , b.p.  $185$ — $195^\circ/25$  mm., are described. C. H.

(A) Dehydration of electrolysable salts of easily oxidisable metals. (B) Purification of magnesium chloride. JOHNSON, MATTHEY & CO., LTD., Asses. of SOC. DE PROD. CHIM. DES TERRES RARES (B.P. 397,479 and 397,481, 22.2.32. Ger., 20.2.31).—Molten chlorides of (A) Ca, Ce, and rare-earth metals, or (B) Mg, are dehydrated and purified from suspended matter by electrolysis at just  $>$  the m.p. with a small current at a voltage  $<$  the decomp. voltage of the salt but  $>$  that of  $\text{H}_2\text{O}$ , and are then kept just molten for 2 hr. without agitation, to allow the impurities to settle. A. R. P.

**Preparation of catalytic materials and of carriers therefor.** H. SPENCE, I. P. LLEWELLYN, and P. SPENCE & SONS, LTD. (B.P. 396,780, 31.10.31).—Aluminous fireclay, indurated by calcination, is extracted with mineral acid, e.g.,  $\text{H}_2\text{SO}_4$  or  $\text{HCl}$ , to remove all or most of the  $\text{Al}_2\text{O}_3$ , and the residual  $\text{SiO}_2$  skeleton is washed, dried, calcined at  $600$ — $700^\circ$ , graded to  $-5$ - to  $+12$ -mesh, soaked in aq.  $\text{Al}(\text{NO}_3)_3$ , and again dried and heated to obtain a catalyst containing  $\text{Al}_2\text{O}_3$  in a highly active form. A. R. P.

**Removing acid constituents from gases.**—See I.  $\text{NH}_3$  from fuel gas. Inert gas.—See II. Solubilising ores containing Fe.—See X. White lead.—See XIII. Fertilisers.—See XVI. Explosive.—See XXII.

#### VIII.—GLASS; CERAMICS.

**Gases in glass.** R. H. DALTON (J. Amer. Ceram. Soc., 1933, 16, 425—432).—An apparatus is described in which gases may be pumped off from a glass at  $1400^\circ$  and analysed. Up to a fraction of 1% of gases has been found and the amount is the lowest with small experimental melts.  $\text{H}_2\text{O}$  is most abundant especially in borosilicate glasses, and the  $\text{CO}_2$  content varies with the basicity of the glass.  $\text{N}_2$ ,  $\text{CO}$ , and  $\text{H}_2$  occur in very small amounts and all oxidised glasses (especially those containing  $\text{As}_2\text{O}_3$ ) contain  $\text{O}_2$ . The refining action of  $\text{As}_2\text{O}_3$  is probably due to  $\text{O}_2$  being liberated into the fine bubbles already present (acting as nuclei), causing them to grow to a size which will escape more easily. Remelting a glass has little effect on the gas content. J. A. S.



**Study of gases in enamelling iron.** R. W. BAKER and J. C. JOUBLANC (J. Amer. Ceram. Soc., 1933, 16, 437—441).—The adsorptive capacity for air,  $N_2$ ,  $H_2$ , and CO of a series of irons containing C, Mn, Si, and Al was measured. The solubility and retention of air and  $N_2$  decreases with decrease in C content, but the solubility of  $H_2$  is unaffected. Increase of Si and Al had little effect on the solubility of gases except in the case of  $N_2$ , which was particularly sol. in the presence of Al. Mn increased the solubility of all gases but CO, with a crit. max. adsorption at 0.25% Mn. A satisfactory Fe is low in C and Mn, but should contain  $\leq$  a certain permissible min. of S, Si, and alloying elements. The further removal of these elements in the open-hearth furnace can be brought about only by an objectionable increase in the  $O_2$  content of the metal. J. A. S.

**"Reboiling" [of enamels on sheet metals].** J. O. LORD (J. Amer. Ceram. Soc., 1933, 16, 442—451).—Recorded results by various experimenters are summarised. J. A. S.

**Peculiarity of sheet-iron acid-resisting cover-coat enamels.** H. E. EBRIGHT and J. E. HANSEN (J. Amer. Ceram. Soc., 1933, 16, 433—436).—Bare spots in the first coat of acid-resisting enamel applied directly on the ground-coat (I) are due more often to "reboiling" of (I) and lack of film strength in the enamel than to "fishscaling" of the (I). The defect is influenced by the reboiling tendency of the base metal. The choice of the kind and amount of "setting up" electrolyte and the application of an intermediate non-acid-resisting enamel will greatly minimise the trouble. J. A. S.

**Effect of organic grinding media on water-soluble silica frits.** H. B. BARLETT and K. SCHWARTZ-WALDER (J. Amer. Ceram. Soc., 1933, 16, 452—454).—The caking of partly  $H_2O$ -sol. frits after grinding is overcome by using org. liquids, preferably polar compounds such as EtOH. J. A. S.

**Leadless glazes for the domestic pottery industry.** L. I. SIVOPLIAS (Ukrain. Chem. J., 1933, 8, 104—109).—A no. of formulæ are given. R. T.

**Water movement in stiff-mud ware and its relation to drying.** E. LOVEJOY (J. Amer. Ceram. Soc., 1933, 16, 405—411).—The rate of loss of  $H_2O$  was determined for test-bars drying from one surface. Movement of  $H_2O$  to the surface takes place until 60% has been eliminated, and thereafter evaporation takes place within the body. The effects of grog, de-airing, flocculation, deflocculation, plasticity, etc. were studied. J. A. S.

**Physical chemistry in porcelain manufacture.** W. J. SUTTON (Lingnan Sci. J., 1933, 12, Suppl., 65—78).—A discussion based on phase-rule considerations. Porcelains with a high mullite content and a min. proportion of undissolved quartz grains have the best mechanical, electrical, and thermal properties. CH. ABS.

See also A., Oct., 1011, Action of electrolytes on kaolin suspensions. 1013, Systems  $ZrO_2$ - $MgO$ ,  $MgO$ - $CaO$ ,  $MgO$ - $BeO$ , and  $CaO$ - $BeO$ . 1020, Adsorption of Ag by glasses. Synthesis of Ca

silicates. 1022, Fe borates. 1024, Determining alkalis in silicates.

#### PATENTS.

**Manufacture of laminated non-splintering glass.** IMPERIAL CHEM. INDUSTRIES, LTD., J. S. B. FLEMING, and A. RENFREW (B.P. 398,189, 7.3.32).—Laminated glass containing an interlayer of polymerised unsaturated compounds, e.g., vinyl acetate, with or without volatile softening agents, is heated, e.g., for 16 hr. at the temp. of incipient softening of the layer, under the application, if desired, of pressure insufficient to cause exudation from between the sheets. L. A. C.

**Stratified bodies, such as strengthened glass.** TRIPLEX SAFETY GLASS CO., LTD., and J. WILSON (B.P. 398,172, 5.3.32. Addn. to B.P. 332,551; B., 1930, 948).—Mono- (I) and di-methyl- (II) -cyclohexanols are used instead of, or together with, cyclohexanol in the prior process; esters (e.g., the oxalate) of (I), solvents or plasticisers (e.g., alkyl phthalates, triaryl phosphates, aromatic sulphonamides,  $CH_2Ph$ -OH, triacetin), and aromatic hydrocarbons (e.g., PhMe,  $C_6H_4Me_2$ ) may also be added. L. A. C.

**Manufacture of bricks, blocks, tiles, etc.** A. WANKLIN (B.P. 398,237, 10.3.32. Addn. to B.P. 341,594).—Before moulding and finally firing, the clay is partly burnt either by calcining at a suitable temp. or by arranging for a (rotary) kiln to deliver batches of dried and fired clay alternately. J. A. S.

**Manufacture of refractory articles [for melting glass, metals, etc.].** H. E. WHITE, Assr. to LAVA CRUCIBLE CO. OF PITTSBURGH (U.S.P. 1,897,183, 14.2.33. Appl., 30.1.30).—A mixture of cryst.  $\alpha$ - $Al_2O_3$  50—60, finely-divided mullite (artificial or calcined sillimanite, cyanite, or andalusite) 30—40, and highly aluminous bond clay 10% is fired at 1650° for 90—150 hr. The ingredients should be as free as possible from glassy material. J. A. S.

#### IX.—BUILDING MATERIALS.

**Influence of magnesium oxide on the strength of mortar.** R. GRÜN (Tonind.-Ztg., 1933, 57, 226; Chem. Zentr., 1933, i, 2596).—The strength of Portland or blast-furnace cement is appreciably diminished by the addition of  $> 5\%$  of  $MgO$ . A. A. E.

**Possible corrosion of iron in ferroconcrete by blast-furnace cement.** P. ERCULISSE (Chim. et Ind., 1933, 29, Spec. No., 1933, 820—821).—Two Fe rods were dipped in freshly mixed Portland cement (A) and 2 others in blast-furnace cement (B) and the e.m.f. in each pair was measured over 2 months. With A it reaches a max. during the height of the setting reaction and then falls to almost zero. With B two maxima occur and the e.m.f. then falls similarly though the later variations are  $>$  with Portland cement. They are not so great as to suggest the likelihood of corrosion. C. I.

**New cements from blast-furnace slags.** P. P. BUDNIKOV and L. GULINOV (Tonind.-Ztg., 1933, 57, 402—403).—Two blast-furnace slags with added anhydrite and burnt dolomite gave strengths in many cases  $>$  Portland blast-furnace cements. The compressive strengths at 6 months of the latter with 15% of Portland



cement clinker were  $>$  in the case of those with 30% of clinker.

T. W. P.

**Concrete and industrial gases.** F. W. FRIESE (Concrete Constr. Eng., 1933, 28, 299—303).—Corrosion of concrete by  $\text{H}_2\text{S}$  is caused by reaction with the free  $\text{Ca}(\text{OH})_2$  in the cement. Oxidation products of  $\text{H}_2\text{S}$  also react with the concrete and with the reinforcements. Losses in strength up to 40—50% have been observed.  $\text{SO}_2$  reacts with the Fe compounds in the cement. Pure  $\text{CS}_2$  is harmless, but when COS is present corrosion occurs. In the presence of moisture  $\text{COCl}_2$  causes disintegration. Examples are given and the mechanism of reactions is discussed. Dense concrete well cured before coming in contact with gases is recommended, to avoid corrosion, but certain protective coatings, details of which are given, may be used.

T. W. P.

**Decay and repair of concrete and masonry dams.** B. HELLSTROM (Struct. Eng., 1933, 11, 210—228).— $\text{H}_2\text{O}$  percolating through dams causes disintegration first through leaching out of free  $\text{Ca}(\text{OH})_2$ , followed by breaking down of hydrated Ca compounds. The effect becomes more serious as the  $p_{\text{H}}$  of the  $\text{H}_2\text{O}$  decreases. For repair the best method is considered to be the application of a sheet of a 1:1 cement-sand mortar covering the whole upstream face.

T. W. P.

**Breakdown of bituminous road-making emulsions in contact with stone.** H. WEBER (Ind. Chem., 1933, 9, 240—243).—The "decomp. val." (Z.W.) of an emulsion is determined by weighing the amount of emulsion retained by graded stone under standard conditions. Different emulsions give different Z.W. for the same stone, and the same emulsion gives different Z.W. for different stones. No fundamental difference was observed in the behaviour of tar and bitumen emulsions. The Z.W. of emulsions do not change with age (5 years). The use of Z.W. in specifications is indicated. An explanation of the breaking of emulsions in contact with stone is offered.

D. K. M.

**Efficiencies of tar-oil components as preservative for timber.** F. H. RHODES and I. ERICKSON (Ind. Eng. Chem., 1933, 25, 989—991).—The lower fractions of a coal-tar creosote "dead" oil, free from tar acids and bases, showed little variation in preservative power and were more effective than higher fractions. Results indicated that no one of the normal components of creosote oil, with the possible exception of  $\text{Ph}_2$ , is the primary cause of preservation. Fractions from water-gas tar were only about 50% as efficient. Chlorination products in general showed an increase in fungicidal power.

C. A. K.

**Rotary kiln heat balance.** [Heat] insulators.—See I. Paints for exhibit buildings. Paint failure on plaster. Radiant energy and paint.—See XIII.

#### PATENTS.

**Manufacture of light concrete and similar materials.** F. E. MARECHAL (B.P. 398,651, 6.10.32).—The usual  $\text{CaO}$  and/or  $\text{MgO}$  base mixture together with colloidal materials is treated in a vat in which high-speed vanes drive the mixture to the walls and cause it to fall over into a central depression. A large contact

surface between the mixture and the atm. above is thus created.

C. A. K.

**[Kiln for] production of artificial lava for use as an aggregate in the production of bituminous concrete or asphalt.** R. H. KING, A. O. CROCKER, B. B. VAN DOMSELAAR, L. N. THOMAS, K. HENDERSON, W. S. ADAMS, and W. A. WESTLEY (CONSTRUCTORA DE CAMINOS "KINGITE," CROCKER, KING & Co.) (B.P. 398,872, 21.3.32).—Material is fed through a slightly inclined rotary kiln into a combustion chamber of truncated cone shape in which it meets the flames from oil or other pressure burners. The melted material discharges through a slot provided.

C. A. K.

**Cement, especially for cementing the middle electrode for sparking plugs.** SIEMENS & HALSKE A.-G. (B.P. 398,767, 20.4.33. Ger., 15.7.32).—For cementing a highly sintered  $\text{Al}_2\text{O}_3$  electrode a cement containing  $\text{Al}_2\text{O}_3$  50—53,  $\text{Al}(\text{OH})_3$  10, quartz powder 15, and Pb-glass powder 5—2 pts. by wt. is used in admixture with dil. aq. Na silicate.

C. A. K.

**Forming massive cementitious bodies.** RIVER-SIDE CEMENT CO. (B.P. 399,101, 2.3.33. U.S., 4.1.33).—A concrete mixture for large work contains Portland cement having a heat of hardening  $< 75$ —80 g.-cal. per g. in 28 days and also a finely-ground cement containing  $3\text{CaO}, \text{Al}_2\text{O}_3 < 12\%$ ,  $2\text{CaO}, \text{Fe}_2\text{O}_3 < 3\%$ ,  $3\text{CaO}, \text{SiO}_2 \geq 10\%$ , and with a function  $\%3\text{CaO}, \text{SiO}_2 + 2(\%3\text{CaO}, \text{Al}_2\text{O}_3) < 45\%$ .

C. A. K.

**Manufacture of building material.** A. M. ZOTTOLI (U.S.P. 1,893,607, 10.1.33. Appl., 19.3.30).—A plastic layer of plaster of Paris or Keene's cement (C) containing  $\text{CaCO}_3$  is laid on a fibrous wallboard foundation in a shallow mould, and to the upper surface of C is applied a glass plate or similar flat object having a design in tartaric acid impressed on it so that the reaction between the acid and the  $\text{CaCO}_3$  produces vein-like cavities by gas evolution in the C surface.

A. R. P.

**Building materials.** E. J. PROPPER (B.P. 398,672, 9.11.32. Switz., 9.11.31 and 14.3.32).—Chippings of natural stones, e.g., basalt, granite, feldspars, etc., are heated in an electric furnace and are either pressed into shape at the sintering temp. (approx.  $1000^\circ$ ) or completely melted at  $1500$ — $1600^\circ$  and cast into the desired form.

C. A. K.

**Elutriation [of cement]. Determining  $\text{H}_2\text{O}$  in wood.**—See I. Mastic agglomerates.—See II.

#### X.—METALS; METALLURGY, INCLUDING ELECTRO-METALLURGY.

**Blast supply, coke charge, and melting efficiency in cupolas.** H. JUNGBLUTH and P. A. HELLER (Arch. Eisenhüttenw., 1933—4, 7, 153—155).—With a const. consumption of coke (A) per 100 kg. of cast Fe melted in the cupola the output (B) in tons per hr. increases linearly with the blast supply (C) and the  $\text{CO} : \text{CO}_2$  ratio in the flue gas (D) remains almost const. When C is kept const. and A is increased the proportion of CO in D increases, and B is inversely proportional to the amount of coke burned to  $\text{CO}_2$ .

A. R. P.



**Basic slags of the open-hearth furnace.** J. BULINA (Chim. et Ind., 1933, 29, Spec. No., 673—678).—Basic open-hearth slags can be classified into three groups according to whether the  $\text{CaO}:\text{SiO}_2$  ratio ( $R$ ) is  $<$ ,  $=$ , or  $> 1.7$ . When  $R = 1.7$  the slag behaves as an ideal solvent for refining the charge, the  $\text{FeO}$ ,  $\text{Fe}_2\text{O}_3$ , and  $\text{Mn}$  oxides therein being in the free state and in the most active form; for a given quantity of  $\text{FeO}$  in the metal bath the corresponding concn. of the  $\text{FeO}$  and  $\text{Fe}_2\text{O}_3$  in the slag depends solely on the temp., falling with rising temp. This type of slag has only a poor desulphurising action. When  $R$  is  $> 1.7$  the slag consists of two phases, viz., (a) liquid  $2\text{CaO}.\text{SiO}_2$  containing dissolved  $\text{Ca}_3\text{P}_2\text{O}_8$ ,  $\text{FeO}$ ,  $\text{MnO}$ ,  $\text{Al}_2\text{O}_3$ , and  $\text{MgO}$ , and (b) a suspension of  $\text{CaO}$ ,  $\text{Fe}_2\text{O}_3$ , and  $\text{MnO}_2$ , probably in combination with one another; the oxides in (b) are in an inactive state and the concn. of  $\text{FeO}$  and  $\text{Fe}_2\text{O}_3$  in the slag is a function of the proportion of free  $\text{CaO}$  in the molten slag and of the temp. This type of slag has a strong desulphurising action, but the loss of  $\text{FeO}$  and  $\text{MnO}$  in the slag increases with increasing  $R$ , and for  $R = 2.3$  is 70%  $>$  when  $R = 1.7$ .

A. R. P.

**Manganese and phosphorus equilibria in the open-hearth furnace in the light of new temperature measurements.** I. C. SCHWARZ, E. SCHRÖDER, and G. LEIBER (Arch. Eisenhüttenw., 1933—4, 7, 165—174; cf. B., 1932, 551).—The equilibrium consts. for the basic open-hearth process have been recalculated from analyses made of the slag and metal at various temp. and with different slags. For the reaction  $\text{FeO} + \text{Mn} \rightleftharpoons \text{Fe} + \text{MnO}$ ,  $K_{\text{Mn}} = [\text{Mn}].(\text{Fe})/(\text{Mn})$  and  $\log K_{\text{Mn}} = -5920/T - 3.32T \times 10^{-4} + 1.778B \times (0.956 - B) + 3.0263$ , and for the reaction  $5\text{FeO} + 2\text{Fe}_3\text{P} + 3\text{CaO} = \text{Ca}_3\text{P}_2\text{O}_8 + 11\text{Fe}$ ,  $K_{\text{P}} = [\text{P}]^2(\text{Fe})^5 \times B^3/(\text{P}_2\text{O}_5)$  and  $\log K_{\text{P}} = 3.62 - 24,000/T$ . The basicity ( $B$ ) of the slag in both reactions is  $0.01(\% \text{CaO} - 0.93 \times \% \text{SiO}_2 - 1.175 \times \% \text{P}_2\text{O}_5)$ ,  $T$  is the abs. temp.,  $[\text{Mn}]$  and  $[\text{P}]$  are the concns. of  $\text{Mn}$  and  $\text{P}$  in the metal, and  $(\text{Fe})$ ,  $(\text{Mn})$ , and  $(\text{P}_2\text{O}_5)$  the concns. of these constituents in the slag;  $[\text{Fe}]$  is taken as unity.

A. R. P.

**Identification of the inclusions in steel.** T. HEPNER and B. OTTA (Chim. et Ind., 1933, 29, Spec. No., 657—659).—The inclusions likely to be found in sheet steel made by the open-hearth process are: (a) particles of slag from the fluxing stage, (b) particles of refractory from the furnace lining etc., and (c) particles of slag from the deoxidation stage. Examination of some sheets under the microscope and of the inclusions in them by chemical analysis after removing the  $\text{Fe}$  with  $\text{I}$  has shown that (c) is the most common inclusion; it usually contains  $> 50\%$   $\text{MnO}$ .

A. R. P.

**Nitrogenisation process of [case]-hardening [steel].** W. LESTMANN (Arch. Eisenhüttenw., 1933—4, 7, 131—139).—On passing  $\text{NH}_3$  at const. speed over  $\text{Fe}$  powder at  $500^\circ$  an equilibrium is eventually set up at which the nitrified  $\text{Fe}$  catalytically decomposes a const. proportion of the  $\text{NH}_3$ ; changes in the gas speed produce changes in the  $\text{N}$  content of the  $\text{Fe}$  and in the  $\text{NH}_3$  decomposed in unit time. The active substance in producing penetration of  $\text{N}$  into steel is the nitride phase of the  $\text{Fe}$ ;  $\text{CrN}$  and  $\text{AlN}$  are more stable and when once formed

take no further part in the nitrogenisation process of nitrifying steels. No state of equilibrium is set up between the surface of the metal and the gas phase, although, as the rate of diffusion of the  $\text{FeN}_2$  into the metal decreases, the amount of  $\text{NH}_3$  decomposed catalytically increases, but is always about 50% less with steel than with soft  $\text{Fe}$ . Penetration of  $\text{N}$  into soft  $\text{Fe}$  proceeds (a) by diffusion from the  $\gamma'$ -phase first formed and (b) by nitrogenisation of the grain boundaries; penetration of  $\text{N}$  into steel ( $\text{C } 0.25$ ,  $\text{Al } 1.1$ , and  $\text{Cr } 1.4\%$ ) proceeds by (b) and by formation of acicular lamellae. Fine segregates due to diffusion and pptn. at the nitrogenising temp. occur only in the innermost layer of the case produced on normalised steel after prolonged treatment with  $\text{NH}_3$ . Addition of substances, e.g.,  $\text{Ni}$ ,  $\text{CaCl}_2$ ,  $\text{CoSO}_4$ ,  $\text{CuSO}_4$ ,  $\text{CaCl}_2$ ,  $\text{NH}_4\text{F}$ , to the treatment chamber does not reduce the time of hardening since once the nitride phase richest in  $\text{N}$  is formed over the surface the diffusion of  $\text{N}$  into the metal is independent of external influences and depends solely on the rate of decomp. of the  $\text{FeN}$  and on the composition and structure of the steel.  $\text{SiO}_2$  gel alone or impregnated with  $\text{CaCl}_2$  in contact with the  $\text{Fe}$  surface does, however, retard considerably the catalytic decomp. of the  $\text{NH}_3$  without affecting the rate of nitrogenisation. A. R. P.

**Tempering of quenched case-hardened steels.** M. CHAUSSAIN (Rev. Mét., 1933, 30, 349—361).—Case-hardened steels containing free  $\text{Fe}_3\text{C}$  in the outer layers soften appreciably at  $> 100^\circ$  owing to the decomp. of the martensite, and the  $\text{Fe}_3\text{C}$  in solid solution tends to reprecipitate at about  $200^\circ$  or after prolonged tempering at  $> 100^\circ$ ; in consequence of these effects the steel becomes brittle and the surface layer tends to scale off, especially if the reprecipitation starts in the middle of the cemented zone and thus produces zones of discontinuity. The presence of  $> 2\%$   $\text{Ni}$  does not prevent these effects, but it tends to refine the structure. With a sufficiently high  $\text{Ni} + \text{Cr}$  content, however, the quenched state is stabilised and tempering has relatively little effect on the properties of the steel. When mechanical parts are subjected during use to a temp. equiv. to tempering, the cementation should be stopped before a hypereutectoid zone containing free  $\text{Fe}_3\text{C}$  is formed at the surface, or a steel with a high  $\text{Ni} + \text{Cr}$  content should be used.

A. R. P.

**Durability of steels for superheater tubes.** A. POMP and W. ENDERS (Mitt. Kaiser-Wilh.-Inst., Eisenforsch. Düsseldorf, 1932, 14, 261—269; Chem. Zentr., 1933, i, 2163).—At  $550^\circ$  a steel containing  $\text{Cr } 0.7$ ,  $\text{Mo } 0.3\%$ , and at  $600^\circ$  one containing  $0.8\%$   $\text{Cu}$  or  $0.3\%$   $\text{Mo}$ , gave the best results.

A. A. E.

**Thick-coated electrodes for steel welding.** A. P. GORACHEV, N. M. NIKITINUKH, and G. J. MARTIANOV (Rep. Inst. Met. Leningrad, 1933, No. 13, 107—121).—A coating containing  $\text{Al}_2\text{O}_3$  1—1.4,  $\text{Fe}_2\text{O}_3$  22—33.6, pyrolusite ( $\text{Mn } 60$ ,  $\text{SiO}_2$  20,  $\text{CaO } 4\%$ ) 50.4—64, and  $\text{CaO}$  13—15% is recommended. The finely-powdered materials are made into a paste with aq.  $\text{Na}_2\text{SiO}_3$  ( $d$  1.16), and the paste is applied 0.5 mm. thick for a 3-mm. rod, or 1 mm. thick for a 4—6-mm. rod. The electrodes, which are dried and heated for 1—2 hr. at  $150$ — $200^\circ$ , can be used for welding steel containing  $> 0.3\%$   $\text{C}$ .

CH. ABS.



**Addenda for the acetylene welding of steel.** E. STREB and H. KEMPER (Autogen. Metallbearb., 1933, 26, 2—8; Chem. Zentr., 1933, i, 1506).—Wire of high C and Mn content is advantageously employed.

A. A. E.

**Effect of absorption of nitrogen and oxygen in the fusion welding [of steel].** E. PIVOVARSKY and W. KLEINEFENN (Arch. Eisenhüttenw., 1933—4, 7, 205—208).—Uncoated (a) and flux-coated (b) welding rods of plain C steel with 0.02—0.1% C (I) and of Ni steel with 0.05—0.15% C and 2—3.5% Ni (II) have been melted by the  $O_2$ - $C_2H_2$  flame (A), by the a.-c. arc (B), and by the d.-c. arc (C), and the O and N contents, the mechanical properties, and the structure of the welds determined. Welds made with (a) rods by methods B and C show the greatest absorption of  $N_2$  and  $O_2$ , whilst those made with (a) rods by method A show less gas absorption than those made with (b) rods by methods B and C, and in all cases method B causes more absorption than method A. Rods of type (II) absorb much less gas than those of type (I). If the amount of  $N_2$  and  $O_2$  absorbed exceeds the solid solubility of these elements in the metal at room temp., subsequent heat-treatment of the welds induces an increase in tensile strength, yield point, and hardness and a decrease in ductility owing to pptn.-hardening effects; the heat-treatment may consist of annealing above A3 and slow cooling, prolonged annealing at 600° and air-cooling, or annealing at 250° after cold-work. Natural ageing at room temp. for prolonged periods also produces a slow hardening effect due to separation of  $N_2$ ,  $O_2$ , and C from supersaturated solid solution. In these respects A welds made with (II a) rods and B and C welds made with (II b) rods are superior to other welds. A. R. P.

**Electrolytic scaling of transformer steel.** O. ESSIN and A. BALABAI (J. Appl. Chem. Russ., 1933, 6, 638—652).—Transformer steel scale (I), containing 70—80% of free Fe, cannot satisfactorily be removed by pickling in  $H_2SO_4$  in contact with Cu, as the voltage of the cell so formed is too small, or by electrolysis in  $H_2SO_4$  or  $Na_2CO_3$  baths, with the steel as the cathode, owing to the low conductivity of (I), with consequent too feeble evolution of  $H_2$  to separate (I) from the metal surface. With the steel as the anode in 30%  $H_2SO_4$ , an abrupt rise in voltage occurs after 1 min., using a c.d. of 7.5 amp. per sq. dm., or after 30 min. using 1 amp., corresponding with passivation of the steel, and with copious evolution of  $O_2$ , which removes (I), leaving a bright, resistant surface. The best results are obtained, using d.c. or a.c. in 16.5% HCl, with a c.d. of 4—5 amp. per sq. dm., during 30 min. Purely chemical methods of scaling are more economical than is the above process.

R. T.

**Relation between the constitution of certain chromium alloys [cast irons] and their rate of dissolution in hydrochloric acid.** P. HERASYMENKO, J. PECH, and F. POBOŘIL (Chim. et Ind., 1933, 29, Spec. No., 649—654).—The rate of dissolution in 2% HCl of cast Fe containing 0.8—2.5% C and 0.35—0.75% Si increases with increase in the Cr content and with rise of quenching temp., i.e., with increase in the proportion of  $\gamma$  in the microstructure. Potential

measurements show that this behaviour is to be attributed to differences in the  $H_2$  overvoltage on the  $\alpha$ - and  $\gamma$ -phases.

A. R. P.

**Use of nitrophenols as etching reagents for iron and iron-manganese carbides.** A. GLAZUNOV and V. PETÁK (Chim. et Ind., 1933, 29, Spec. No., 691—693).—Substitution of *p*-nitrophenol for picric acid in Le Chatelier's etching reagent yields a reagent which has no action on pearlite but colours cementite (C) red to violet with patches of green, due to the varying C content from the centre towards the periphery of the C areas caused by a stepped transformation of the  $Fe_3C$ . With high Mn content a homogeneous green colour is imparted to C, showing the stabilising effect of the Mn.

A. R. P.

**Permanent magnet steels on the basis of precipitation-hardening.** W. KÖSTER (Stahl u. Eisen, 1933, 53, 849—856).—Steels for permanent magnets with better properties than those of martensitic plain-C steels are now made of binary or ternary alloys which can be pptn.-hardened. The binary alloys suitable are those with a closed  $\gamma$ -field and having a limited solubility of the alloying elements (A), which increases with rise in temp.; the ternary alloys may be of two types: (a) those in which the solubility of at least one of the A increases with rise in temp., and (b) those in which the limited solubility is caused by the simultaneous presence of both of the A. In group (a) the best magnetic properties are shown by steels containing Co and W or Mo, which have coercivities of 60—350 oersteds with a high remanence; the alloys can be worked hot and readily machined. The best alloys in group (b) are the Ni-Al-Fe alloys, which have coercivities of > 600 oersteds; the alloys can be used only in the cast state and can be worked only by grinding. The structure and magnetic properties of typical alloys of these groups are discussed with reference to micrographs and curves.

A. R. P.

**Tests of regular and stabilised 18 : 8 [Cr-Ni-Fe] alloy welds in sulphite[-cellulose] liquor.** ANON. (Paper Trade J., 1933, 97; T.A.P.P.I. Sect., 105—110).—Welded specimens of these alloys having also high C (A), low C (B), or Ti + low C (C) contents have been tested by exposure to sulphite liquor (I), to  $H_2SO_4$ - $CuSO_4$  solution (II), and to high temp. (650°). The degree of corrosion was determined by loss in wt., microscopical examination, torsion tests, and measurement of changes in electrical resistance. B and especially C alloys can be employed in the welded condition without suffering intergranular corrosion by (I); A alloys should always be heat-treated after welding. Exposure to boiling (II) provides a relatively rapid method of determining probable resistance in service.

H. A. H.

**Corrosion phenomena in high-pressure boilers.** K. HOFER (Stahl u. Eisen, 1933, 53, 925—929).—Three general types of corrosion are noted: (a) flat depressions caused by the  $p_{H_2}$  of the  $H_2O$ , (b) local pits due to a high  $O_2$  content of the  $H_2O$ , and (c) scaling due to the direct action of the steam on the steel. These effects may be accentuated by mechanical action in the boiler (B), by the presence of stresses in the steel and irregu-



larities in its structure, by constructional faults especially those causing disturbances in the  $H_2O$  flow, by incorrect prep. of the feed- $H_2O$  (I), and by the formation of *B* scale and foaming of the  $H_2O$ . Examples of the action of these faults are shown and discussed. To prevent their occurrence the (I) must be carefully purified and degassed; it must have the min. alkalinity and contain a certain amount of protective salt depending on the nature of the  $H_2O$ , the performance of the *B*, the pressure, and the working conditions. In building the *B* cold-work must be studiously avoided to prevent internal stresses, and the working surface should be of such a nature as to prevent adherence of gas or steam bubbles. A. R. P.

**Corrosion in sulphonators.** ANON. (Chem. Met. Eng., 1933, 40, 409).—The rate of corrosion (figures are given) of metals during the sulphonation of neatsfoot oil is in the following order (most resistant first): Ni (*A*), monel metal (*B*), Ni-resist (*C*), Pb (*D*), Cr-Ni-Fe (*E*), cast Fe (*F*). For castor oil the order is: *B*, *E*, *A*, *C*, *F*, *D*. D. K. M.

**Reduction of corrosion in water-pipe.** E. S. HOPKINS (Ind. Eng. Chem., 1933, 25, 1050).—Corrosion of Fe pipes for  $H_2O$  can be checked by treating with CaO to maintain  $pH$  7.7. E. S. H.

**Continuous measurement of slight corrosion [of steels] accompanied by evolution of gas.** A. PORT-  
EVIN, E. PRÉTER, and L. GUITTON (Rev. Mét., 1933, 30, 362—365).—The specimen (I), the lower side of which is coated with wax and the upper side brightly polished, is suspended horizontally by means of a glass thread from the beam (II) of a balance and a watch-glass is fixed to the thread a few cm. above (I) so as to catch the bubbles of gas; the whole is immersed in the corroding medium (III) and the movement of (II) observed through a lens. The vol. of  $H_2$  evolved is thus measured by weighing the amount of (III) displaced and can be converted into loss in wt. if the composition of (I) as well as the relative rates at which its constituents dissolve are known. During the first 10—15 min. of the test a small loss of  $H_2$  occurs due to capillary phenomena at the surface of the metal; these effects, however, become of relatively minor importance with increasing period of immersion. The results obtained with steels in dil.  $H_2SO_4$  are shown graphically. A. R. P.

**Behaviour of new rust-protecting materials under electrical, thermal, and vibrational conditions.** W. BECK (Gas- u. Wasserfach, 1933, 76, 591—598).—The behaviour of protective coatings on underground steel or Fe pipes in various soils can be determined by measuring the change in c.d. with time when the coated pipe (*A*) is immersed as anode in the soil near a Sn-plate cathode (*B*) and a const. potential of, e.g., 3 volts is maintained between *A* and *B*. Dynamic tests can be made in the same way except that *A* is rotated in an alternating-bending machine and the soil is containing in an Al pipe (*C*) surrounding *A*, measurements being made of the current passing from *A* to *C* under const. potential; the temp. can be varied by cooling *A* internally. Results of tests made with numerous bituminous, rubber, and paraffin coating mixtures are shown graphically and in tables. A. R. P.

**Stability of trichloroethylene; examination of its metal corrosion effects.** ANON. (Chem. Trade J., 1933, 93, 227—229).—The corrosive action of  $CCl_4$  (I) and  $C_2HCl_3$  (II) on mild steel (*A*), cast Fe (*B*), staybrite (*C*), Cu, gunmetal, monel (*D*), brass, Zn (*E*), Sn, Al (*F*), and Pb has been determined in the presence and absence of  $H_2O$  and in the liquid and gaseous phase. When wet (I) is refluxed over the metal only *C*, *D*, and *F* are unattacked, but with (II) under these conditions only *A*, *B*, and *E* are attacked. Addition of an alkylamine prevents attack of *A* and *B* in the latter case. It follows that under normal degreasing conditions (II) has practically no effect on any of the common metals since the commercial solvent always contains an alkylamine and therefore remains alkaline during use. Photochemical decomp. cannot occur owing to the type of vat used in the degreasing process. A. R. P.

**Effect of insufficient pickling on subsequent galvanising.** V. A. WARDELL (Ind. Chem., 1933, 9, 233—234; cf. Edwards, B., 1933, 390).—The production of "skinned" coatings (I) in galvanising is associated with soft, fully annealed sheets. Longitudinal raised streaks (II) on (I) are probably due to particles of Zn-Fe alloy distributed through the coating. (II) have been found on galvanised sheets with a good normal appearance. It is suggested that for a low-C surface with large ferrite grains unless these are deeply pitted the Zn-Fe alloy is neither so adherent nor so coherent as for a fine-grained or deeply etched surface, and tends to disperse through the adjacent Zn, thus reducing fluidity and interfering with crystallisation. D. K. M.

**Preparation and etching of galvanised wire for metallographic examination.** L. HAJDA and S. POPOFF (Chim. et Ind., 1933, 29, Spec. No., 700—704).—The wire is mounted in a piece of ebonite and a transverse section is polished by means of an EtOH suspension of MgO on a waxed disc. The best etching reagent is a mixture of 1 pt. of a 4% solution of  $HNO_3$  in  $Ac_2O$  and 10 pts. of a 1:1:1 vol. mixture of MeOH, AcOH, and  $iso-C_5H_{11}OH$ . This reagent serves to reveal the constituents of the Zn layer and to distinguish between deposits produced electrolytically and by hot-dipping. A. R. P.

**Determination of the thickness and quality of the zinc deposit on galvanised iron wires.** A. GLAZUNOV (Chim. et Ind., 1933, 29, Spec. No., 688—690).—A piece of wire 20—30 cm. long is coated with wax except for 3—4 cm. a short distance from the lower end. The wire is immersed inside a Pt cylinder in conc. aq.  $ZnSO_4$  so that the unwaxed portion is completely covered, and connected with the positive pole of a battery, the Pt being connected with the negative pole through an ammeter and a voltmeter being connected across the cell. During the anodic dissolution of the outer layer of pure Zn (*A*) the voltage remains const., then slowly rises as the layer of Fe-Zn compounds (*B*) is dissolved, and finally again becomes const. when the Fe core is exposed. The thickness of *A* and *B* can be calc. from the dimensions of the wire, the amperage, and the time taken at each stage of the dissolution. A. R. P.



**Removal of the coloured layer (brass) from plated iron-ware cuttings.** V. A. CHEIFETZ (J. Appl. Chem. Russ., 1933, 6, 653–664).—The brass-coated Fe is made the anode in a solution containing 16 g. Cu [as  $\text{Cu}(\text{NO}_3)_2$ ] per 100 c.c., when passivation of Fe occurs, with increase of its solution potential above that of Cu, which is transported to the cathode. The temp. should be  $> 65^\circ$ , and the c.d.  $> 4$  amp. per sq. dm. Max. conductivity is obtained using  $\text{M}-\text{Cu}(\text{NO}_3)_2$ . The contents of spent baths are regenerated by diluting the liquor, when  $\text{Fe}(\text{OH})_3$  separates out, filtering, and pptg. Cu as  $\text{Cu}(\text{OH})_2$  by addition of  $\text{ZnO}$ , and dissolving the  $\text{Cu}(\text{OH})_2$  in  $\text{HNO}_3$ . Zn is pptd. as  $\text{ZnCO}_3$  and  $\text{NaNO}_3$  is crystallised out from the filtrate. R. T.

**Action of sulphur on certain metals [copper and silver].** R. DUBRISAY (Chim. et Ind., 1933, 29, Spec. No., 631–632).—When polished Ag or Cu sheets are suspended below pure dry S in vac. they slowly become covered with a brown to black sulphide film. Ag is also blackened by contact with dry  $\text{C}_6\text{H}_6$  containing 0.1 mg. S per 100 c.c. The active agent in the blackening of these metals therefore appears to be uncombined S. A. R. P.

**Flotation with a miniature machine.** T. MAYEDA (Tech. Rep. Tôhoku, 1933, 11, 93–112).—Flotation tests have been made in a miniature machine in which one variable at a time could be changed. Using a synthetic mixture of quartz and chalcopryrite, the results indicate that, as regards particles of a size between 150- and 450-mesh, the finer is the particle the more readily it floats; each size of particle has an optimum amount of oil for obtaining max. recovery, and the recovery increases linearly with the speed of the impeller, with rise in temp. to a max. at  $40^\circ$ , and with the pulp ratio to a max. at 2:1. With charges of very uneven particle size better recoveries are obtained by adding the oil in two stages. A. R. P.

**Magnesium alloy protection by selenium and other coating processes.** II. G. D. BENGOUGH and L. WHITBY (Inst. Metals, Sept., 1933. Advance copy, 4 pp.; cf. B., 1932, 511).—Visual examination or the loss in wt. fails to indicate the loss in mechanical properties of Mg alloys when subjected to the action of sea- $\text{H}_2\text{O}$ . Of all the painted specimens of Elektron AZM alloy, those coated with Se suffered the least loss of elongation. C. A. K.

**Corrosion-fatigue characteristics of an aluminium specimen consisting of two crystals.** H. J. GOUGH and D. G. SOPWITH (Inst. Metals, Sept., 1933. Advance copy, 16 pp.).—An alternating torsion test showed no evidence that more intensified corrosion at the crystal boundary would lead to intercryst. failure, and that the boundary had extremely little effect on the stress distribution, as indicated by the slip. Small pits had no influence on the failure of the specimen. C. A. K.

**X-Ray inspection of aluminium alloy welds.** N. C. HYPHER (Metallurgia, 1933, 8, 145–146, 148).—X-Ray photographs of welds in Al and its alloys are given showing the characteristics of good and faulty welds.  $\text{Al}_2\text{O}_3$  inclusions are not revealed by this method, which is also unsuitable for examining spot-welds. A. R. P.

**Determination of small quantities of zinc in aluminium.** M. I. SHUBIN (Tzvet. Met., 1932, 147–162).—The metal (5–10 g.) is dissolved in 20% aq. NaOH (125–250 c.c.); the solution is diluted to 250–400 c.c., filtered, and the filtrate treated with 10–15 c.c. of 20% aq. NaOH which has been saturated with  $\text{H}_2\text{S}$ . After boiling, the insol. sulphides are collected, dissolved in 6N- $\text{H}_2\text{SO}_4$  (25–32 c.c.), boiled to remove  $\text{H}_2\text{S}$ , and oxidised with  $\text{H}_2\text{O}_2$ . The solution is made slightly alkaline with aq.  $\text{NH}_3$ , excess of  $\text{NH}_3$  is removed by boiling, 1–2 c.c. of 25% aq.  $\text{NH}_3$  is added, the solution is filtered, and the filtrate is electrolysed after addition of 4–5 c.c. of 25% aq.  $\text{NH}_3$ . CH. ABS.

**Treatment of sulphide ore from the Lancefield Gold Mine, Beria, W. Australia, by flotation.** W. G. CLARKE and B. H. MOORE (Kalgoorlie Bull., Sch. Mines W. Australia, 1932, No. 7, 7–11).—Methods involving flotation and cyanidation have been studied. CH. ABS.

**Treatment of concentrates at Nikitovski Mercury Combine.** V. A. VANYUKOV (Tzvet. Met., 1932, 136–146).—Concentrates (22–44% Hg) afforded 99% recovery by the use of a reverberatory furnace. CH. ABS.

**Thermal conductivity of metals in relation to their use in the chemical industry, especially the manufacture of acids and explosives.** Z. STEFAN (Chim. et Ind., 1933, 29, Spec. No., 964–975).—The applicability of various metals and alloys for numerous types of plant is discussed in respect of their thermal conductivity, their resistance to corrosion, and their mechanical properties, rules governing the conductivity being given. The heat transfer of air and vapours and of boiling, moving, and stationary liquids across Cu and Pb partitions has been calc. The working of these metals and alloys for the construction of plant is considered. W. J. W.

**Physical properties of commercial thorium.** J. G. THOMPSON (Met. & Alloys, 1933, 4, 114–118).—Attempts to determine the m.p. of electrolytic Th (A) and Th produced by reduction of  $\text{ThCl}_4$  with Ca (B) gave unsatisfactory results owing to the high reactivity of the metal and to the evolution of vapours at the m.p.; the best vals. obtained were (A)  $1680^\circ$  and (B)  $1730^\circ$ . Th diffuses readily into  $\text{ThO}_2$ , and  $\text{ThO}_2$  into Th, thereby raising the m.p. The lattice parameter of (A) is  $5.091 \pm 0.004 \text{ \AA.}$ , scleroscope hardness 28,  $d$  11.1–11.49,  $d_{\text{calc.}}$  11.61, electric resistivity at  $20^\circ$   $18.6 \times 10^{-6}$  ohm-cm., temp. coeff. of resistance  $3.8 \times 10^{-3}$ , coeff. of linear expansion (20– $300^\circ$ )  $12.1 \times 10^{-6}$ , e.m.f. against Pt  $-0.13$ ,  $-0.53$ ,  $-0.21$ ,  $+0.87$ ,  $+5.42$  mv. at  $100^\circ$ ,  $500^\circ$ ,  $700^\circ$ ,  $900^\circ$ , and  $1300^\circ$ , respectively. A. R. P.

**Practical "test" manipulation. Handling the cupellation furnace.** C. C. DOWNIE (Chem. & Ind., 1933, 777–778).—Modern cupellation practice for the recovery of Ag, Au, and the Pt metals from Pb is briefly described. Addition of As to the Pb before cupellation and skimming of the speiss formed removes all the Ir and thus prevents it from entering the bed of the "test." A. R. P.

**Heat-treatment of "standard silver."** H. O'NEILL, G. S. FARNHAM, and J. F. B. JACKSON (Inst.



Metals, Sept., 1933. Advance copy, 11 pp.).—The reheating of quenched standard Ag (7.25% Cu) to induce progressive pptn. gives a hardness curve passing through a max. which corresponds with a min. in the Meyer *n*-val. curve, and the larger grain size is accompanied by a fall in hardness. X-Ray spectrograms after reheating at 200°, 300°, and 500° for 30 min. indicated a normal progressive pptn. of the Cu constituent with rise in temp. Care should be taken with the surface prep. and polishing, and etching should be avoided. Heat-treatment in high vac. is recommended. C. A. K.

**Treatment of slimes from Tasmania mine, Beaconsfield, Tasmania.** W. G. CLARKE and B. H. MOORE (Kalgoorlie Bull., Sch. Mines W. Australia, 1932, No. 7, 15–17).—Roasting and cyanidation gave the best results. CH. ABS.

**Treatment of cyanide sand residues from the White Hope Gold Mine, Hampton Plains.** W. G. CLARKE and B. H. MOORE (Kalgoorlie Bull., Sch. Mines W. Australia, 1932, No. 7, 22–25).—Concn. of sulphide minerals by strakes or tables, or amalgamation, was unsuccessful. Leaching, by percolation with CN' solution, of the finely-ground material is recommended. The use of saline well-H<sub>2</sub>O is discussed. CH. ABS.

**Cyanidation of tailings.** W. G. CLARKE and B. H. MOORE (Kalgoorlie Bull., Sch. Mines W. Australia, 1932, No. 7, 18–19).—Tests with tailings containing 4.3 dw. Au per 2000 lb., using H<sub>2</sub>O of *p*<sub>H</sub> 8.0, are recorded. CH. ABS.

**Treatment of tailing from the Floater gold mine, Ravensthorpe.** W. G. CLARKE and B. H. MOORE (Kalgoorlie Bull., Sch. Mines W. Australia, 1932, No. 7, 12–14).—Recommendations are made; the tailing contains 5.0 dw. Au per 2000 lb., and 0.13% Cu. CH. ABS.

**Evaluating gold in certain placers by microscopy.** A. L. CRAWFORD (Min. & Met., 1933, 14, 372–374).—Since the Au in placers always occurs as minute nuggets a fair appraisal of the val. of such a deposit can be obtained only by working on large samples; the ordinary quantities taken for assay by the usual methods give very erratic results. A sample of several lb. is panned to give a small concentrate, and this is examined under the microscope, the specks of Au being picked out and weighed. A. R. P.

**Micro- and spectro-assay of noble metals.** C. PETERS (Metallwirt., 1933, 12, 17–19; Chem. Zentr., 1933, i, 1659).—Ag, Au, Ru, Rh, Pd, Os, Ir, and Pt are determined, without chemical separation, by "micro-dosimastic accumulation" in a Pb regulus. Most of the Pb is then removed and the residue examined spectro-analytically. A. A. E.

**Rapid zinc electroplating from hot cyanide solutions.** ANON. (Synth. and Appl. Finishes, 1933, 4, 184–189).—A description of existing practice, with details of control and analysis of the bath. E. S. H.

**Removal of metallic deposits by high-frequency currents.** J. K. ROBERTSON and C. W. CLAPP (Nature, 1933, 132, 479–480).—The application of a high-frequency field by means of external wire electrodes

removed wall deposits near the electrodes. No removal occurs unless a gas or vapour is present to carry a discharge. With H<sub>2</sub> and O<sub>2</sub> removal is much more rapid than with N<sub>2</sub>; the inner surface of the discharge tube is markedly changed. L. S. T.

**Wear in the polishing of plated and other surfaces.** O. F. HUDSON (Inst. Metals, Sept., 1933. Advance copy, 6 pp.).—The rate of wear (I) of platings when polished with MgO on wet parchment showed no consistent relationship with the hardness. The (I) of "pure" Pd was twice that of "pure" Pt when considered as loss of thickness, and was greater when the metals were in massive form than when plated. The (I) of the precious-metal coatings was approx. that of Ni plating, and Ag was definitely the least resistant to wear. C. A. K.

**Use of the microscope in the study and control of electrolytic deposits.** A. PORTEVIN and M. CYMBOLISTE (Rev. Mét., 1933, 30, 323–348).—Methods of testing the quality of Ni, Cu, and Cr plates on steel are described with especial reference to the use of the microscope in detecting pores, pits, cracks, excrescences, and irregular thickness or distribution of the plate, 74 photomicrographs of characteristic good and bad plates being shown. Inclined sections through the deposits give most information on the structure, and the Erichsen test provides a rapid method for determining the adherence of the plate to the base metal. The effect of variations in the composition of the bath and in the plating conditions on the character of Ni plates is illustrated. A. R. P.

**Action of C<sub>2</sub>H<sub>2</sub> on Cu etc.**—See XI. Fe. Enamels for Fe etc.—See VIII. Cement from blast-furnace slag. Corrosion of Fe in ferroconcrete.—See IX. Coatings for underground pipes, sheet Cu, and Parkerised etc. metal. Metal preservatives.—See XIII. Tinning of conductors.—See XIV.

See also A., Oct., 1003, Electrolytic Cr deposits. ZrW<sub>2</sub>. 1006, Diffusion in Au–Ni, –Pd, and –Pt pairs. Analysis of alloys of Hg with Ag, Au, and Sn. 1007, Constitution of alloys. Systems Fe–Si, Be–Cu, Zn–Cd, Pb–Sb, Ag–Li, and Fe–Co–Mn. Solubility of MgZn<sub>2</sub>, Cu, and Si in solid Al. Si–Al alloys. Hardening transformation in Mn steels. Equilibrium A3 and Acm points in pure C steels. Influence of heavy metals in Al alloys. 1008, Fe–Mn alloys. Ternary alloys of Ti with Pb, Cd, and Sn. Heats of mixing of Fe–Ni–Co alloys. 1013, Oxidation of liquid Fe by steam. 1016, H<sub>2</sub> overvoltage of Pb and Pb–Sb alloys. Anodic behaviour of Fe. 1017, Atm. corrosion of Mg. 1019, Spongy Zn electrodeposits. Cr-plating. 1020, Prep. of colloidal Au solution. 1022, Corrosion of Fe. 1024, Potentiometric Ag titration. 1025, Macroelectro-analysis of Co, Zn, Cd, Ag, and Hg. 1026, Microscopical investigation of ores and metals. 1027, Stainless steel high-pressure ultra-filter. 1031, Fe ore deposits.

#### PATENTS.

**Annealing process.** F. C. KELLEY, ASSR. to GEN. ELECTRIC Co. (U.S.P. 1,894,825, 17.1.33. Appl., 1.5.30).—A steel strip is heated and cooled in H<sub>2</sub>; within the main tube of the furnace and cooler an additional tube



surrounding the strip is provided in the neighbourhood of the boundary between the heating and cooling zones.

B. M. V.

**[Steel] chemical apparatus and method of lining same.** R. L. DUFF, Assr. to STANDARD OIL DEVELOPMENT Co. (U.S.P. 1,883,630, 18.10.32. Appl., 27.12.26).—Steel distillation and oil-cracking apparatus is lined by arc- or gas-welding with a Cr-Fe alloy, *e.g.*, that containing Cr 16—18, Si 1.25—1.75, and C 0.1%. A. R. P.

**Alloy steels.** ELECTRO METALLURG. Co., Assees. of R. FRANKS (B.P. 397,646 and Addn. B.P. 397,717, [A] 2.7.32, [B] 12.11.32. U.S., [A] 29.7.31, [B] 10.12.31).—Claim is made for steel containing (A) Cr 15—35, C 0.1—2, and N 0.2—0.4%, or (B) Cr 15—35, Ni 5—35, C < 0.1, and N < 0.1%. The N is added preferably as Cr nitride.

A. R. P.

**Wear-resisting ferrous alloys.** A. W. GREGG and R. H. FRANK, Assrs. to BONNEY-FLOYD Co. (U.S.P. 1,894,819—20, 17.1.33. Appl., [A] 24.3.31, [B] 4.8.32).—Alloys suitable for application to softer steel by welding comprise: (A) Cr 10—20, W 10, Mo 1, V 1, Ni 2, C 1.5—4.5%; (B) Cr 11—19 (14—16), W 12—20 (15—18), V 0.5—2.0 (0.75—1.25), Ni 21—31 (24—26), C 2.00—3.50 (2.5—3.0)%, the balance in each case being Fe and traces only of impurities.

B. M. V.

**Metal [steel]-cleaning composition.** H. C. MOUTGEY, Assr. to GEN. MOTORS RESEARCH CORP. (U.S.P. 1,897,813, 14.2.33. Appl., 1.8.29).—The surface is sprayed or brushed with a mixture of 85%  $H_3PO_4$  (1), EtOH (1), and a  $H_2O$ -sol. oil solvent ( $\frac{1}{4}$ —2 pts.), preferably an ether of a polyglycol.

A. R. P.

**[Inhibitor for] acid pickling baths [for steel].** IMPERIAL CHEM. INDUSTRIES, LTD., A. MACARTHUR, and A. J. HAILWOOD (B.P. 397,553, 26.2.32).—A long-chain alkyl quaternary  $NH_4$  compound, *e.g.*, cetylpyridinium bromide, is claimed.

A. R. P.

**Manufacture of corrosion-resistant coatings on metal articles.** (Miss) A. THIELMANN (B.P. 397,267, 24.5.32. Ger., 29.12.31).—The articles, *e.g.*, pipes or cables (I), are coated with a mixture of oxidised petroleum bitumen, vaseline, and paraffin wax containing asbestos and/or powdered shale as filler. Jute or felted wool bands impregnated with the bituminous mixture (without filler) may be used as bandages for (I).

A. R. P.

**Solubilisation of metal values in oxidised ores [of copper and/or nickel] containing iron.** MEYER MINERAL SEPARATION Co., Assees. of R. F. MEYER (B.P. 396,933, 3.8.32. U.S., 4.5.32).—Oxidised or roasted sulphide ores of Cu and Ni are reduced with powdered coal at  $\geq 750^\circ$  to reduce most of the  $CuO$  to metal and the  $Fe_2O_3$  to  $FeO$ . The roasted product is then treated at  $150$ — $300^\circ$  with a regulated quantity of  $Cl_2$  and air to convert the Cu and Ni into chlorides and the  $FeO$  (which acts as a Cl carrier) into  $Fe_2O_3$ . Small quantities of  $NaCl$  or  $CaCl_2$  may be added to the charge in the roasting or reducing operation to convert any  $FeSO_4$  formed into  $FeCl_2$ .

A. R. P.

**[Nickel-copper-silver alloy] contact material.** E. W. ENGLE, Assr. to FANSTEEL PRODUCTS Co., INC. (U.S.P. 1,883,650, 18.10.32. Appl., 22.6.31).—An alloy of Ag 65, Cu 30, and Ni 5% is claimed.

A. R. P.

**Purifying zinc.** NEW JERSEY ZINC Co., Assees. of P. McL. GINDER (B.P. 397,061, 27.3.33. U.S., 20.12.32).—In the fractional-distillation method of refining Zn (cf. B.P. 392,361; B., 1933, 634), a series of retorts each with its own reflux column is connected with a common condenser heated above the m.p. of Zn so that the metal collects in a liquid form.

A. R. P.

**Soldering fluxes.** C. L. BARBER, Assr. to KESTER SOLDER Co. (U.S.P. 1,882,734—5, 18.10.32. Appl., [A] 16.12.29, [B] 17.3.30).—Claim is made for (A) a mixture of an org. amide, *e.g.*, urea or  $NH_2Ac$ , and an amine hydrochloride, *e.g.*, the betaine,  $NH_2Me$ , or  $NH_2Ph$  compounds, or (B) the product obtained by fusing  $ZnCl_2$  and stearic acid with or without oleic acid.

A. R. P.

**Winning of highly refractory carbides [on wires etc.].** N. V. PHILIPS' GLOEILAMPENFABR. (B.P. 397,372, 19.1.33. Ger., 29.1.32).—A C filament is heated electrically in an atm. of a volatile compound of the metal the carbide of which is desired; *e.g.*, for Si, Ti, Zr, Hf, Ta, or Nb carbide the corresponding chloride is used.

A. R. P.

**Electrolytic production of magnesium.** JOHNSON MATTHEY & Co., LTD., Assees. of SOC. DE PROD. CHIM. DES TERRES RARES (B.P. 396,820 and 396,827, [A] 17.2.32, [B] 19.2.32. Ger., [A] 17.2.31, [B] 19.2.31).—(A) The Fe cathode which protrudes through the side or bottom of the cell is cooled by passing air through it at such a speed that the tip from which the molten Mg rises is kept at  $< 700^\circ$ . (B) The Mg produced from a cathode as in (A) is allowed to rise into a container immersed in the centre of the cell until the layer is up to 10 cm. thick; in this way opportunity is given for suspended impurities in the Mg to settle out.

A. R. P.

**Combustion control in furnaces.**—See I. Reheating of steel etc.—See II. Phosphates [from ferro-phosphorus].—See VII. Metal-melting refractories.—See VIII.

## XI.—ELECTROTECHNICS.

**Insulation substances for the cable industry, with special reference to cellulose esters.** P. NOWAK (Angew. Chem., 1933, 46, 584—587).—Compared with oil-impregnated paper, cellulose acetates have the higher dielectric const. (I) and dielectric losses (II), but these are less affected by rise in temp., the breakdown voltage is higher, and the hygroscopicity (III) lower. The acetates are brittle but addition of plasticisers overcomes this defect. Mixed esters have lower (I) and (II) than simple acetates, and these vals. fall with rising temp.; (III) is also lower.

A. G.

**Extraction of humic acids. Decomp. of oil emulsions. Transformer and insulating oils.**—See II.  $HNO_3$  concn. control. Determining alkalis in  $CaO$  etc. Dehydration of salts. Pure  $MgCl_2$ .—See VII. Transformer and magnet steels. Determining thickness of Zn on galvanised Fe. Plated Fe-ware. Zn-plate. Removal of metallic deposits. Wear of plated surfaces. Electro-deposits.—See X. Finishes for ebonite.—See XIII. Electrical properties of rubber. Insulated conductors.—See XIV. Testing safety fuse and explosives.—See XXII.



See also A., Oct., 1003, Electrolytic Cr deposits. 1016,  $H_2$  overvoltage of Pb and Pb-Sb alloys. Anodic behaviour of Fe. 1019, Spongy Zn electrodeposits. Cr-plating. 1022, Sb electrode for  $p_H$  determinations. 1022, Electrode potential of  $Cl_2$ . 1024, Potentiometric Ag titration. 1025, Macro-electro-analysis of Co, Zn, Cd, Ag, and Hg. Electric furnaces. 1026, Mo resistance furnace. 1026, Extremely low temp. High-temp. X-ray camera. 1027, Electric sieve. Electrode pair for rapid electro-metric analysis.

## PATENTS.

**Manufacture of electrolytes for dry cells which require filling with fluid.** J. J. PÁLA (B.P. 398,638, 2.9.32. Czechoslov., 15.6.32).—Exciting salts are mixed with starch or starch-containing substances, *e.g.*, gluten, and  $H_2O$ , kneaded, comminuted, and dried at about  $60^\circ$ . J. S. G. T.

**Electrolytic cells [condensers].** R. JAHRE [R. JAHRE, SPEZIALFABR. F. KONDENSATOREN] (B.P. 399,037, 17.11.32).—A construction of cells in which electrolyte is prevented from creeping along the foils is claimed. J. S. G. T.

**Copper oxide rectifier.** G. F. MARTIN, JUN., ASSR. to WESTINGHOUSE ELECTRIC & MANUFACTURING CO. (U.S.P. 1,901,563, 14.3.33. Appl., 16.9.26).—A Cu blank coated with  $Cu_2O$  and a superposed layer of CuO by heating in air or an atm. containing  $O_2$  is quenched in a mineral oil (gasoline), vegetable oil, EtOH, etc., whereby the CuO is reduced to Cu. J. S. G. T.

**Devices [screens] for electrolytic separation of substances from liquid.** K. KAISER (B.P. 398,751, 25.3.33. Fr., 26.3.32).—Screening elements arranged near the electrodes are provided with elastic closing members, *e.g.*, of rubber, adapted to be inflated for closing the passages and preventing return of decomp. products and interrupting the electrolysis. J. S. G. T.

**Electrical separation of suspended particles from gaseous fluids.** LODGE-COTTRELL, LTD. From SIEMENS-LURGI-COTTRELL-ELEKTROFILTER-GES.M.B.H. F. FORSCHUNG U. PATENTVERWERTUNG (B.P. 398,724, 3.2.33).—The polarity of the electrodes is changed at intervals, *e.g.*, of  $\frac{1}{2}$  sec. J. S. G. T.

**[Electrically] detecting, controlling, and analysing gases, mixtures of gases, smokes, and dusts suspended in gases.** P. MALSALLEZ (B.P. 398,722, 1.2.33. Fr., 14.5., 7.11., and 16.12.32).—The current formed from charges produced by subjecting the gas to the ionising action of radioactive material in an electric field is amplified and caused to operate a signalling or other device. J. S. G. T.

$C_2H_2$ .—See II. Battery acid.—See VII. Cement for sparking plugs.—See IX. Corrosion-resistant coatings [for cables]. Carbides [on wires]. Mg.—See X. Sterilisation.—See XIX.

## XII.—FATS; OILS; WAXES.

**Free fatty acid content of herring-meal fat.** A. MOEN (Tidsskr. f. d. norske Landbruk, 1933, 40, 153—157).—Herring meal (I) of low  $H_2O$  content keeps

better than that of high. Quality (II) can sometimes be judged on  $NH_3$  content (III), but the (III) of well-dried (I) may be low. In such cases the content of free fatty acids, normally about 5% of the fat, gives a good indication of (II), being as high as 40% in samples where much decomp. has occurred. NUTR. ABS. (m)

**Determination of unsaponifiable matter in fats.** V. VESELÝ and L. K. CHUDOŽILOV (Chim. et Ind., 1933, 29, Spec. No., 1048—1050).— $Et_2O$ -extraction methods must be employed for materials containing waxy matter, as the low results obtained by the Spitz-Hönig method (extraction with light petroleum) appear to be due to loss of alcohols (aliphatic and sterols) by dissolution in the alcoholic soap solution. The more convenient Spitz-Hönig method gives satisfactory results where hydrocarbons only are concerned, *e.g.*, separation of admixed mineral oil. VIZERN remarks that 6 extractions of lanoline with light petroleum yielded as much unsaponifiable matter as 3 extractions with  $Et_2O$ . E. L.

**Gross examples of rancidification of soaps.** F. WITTKA (Allgem. Oel- u. Fett-Ztg., 1933, 30, 381—385).—Abnormally rapid rancidification and discoloration of a certain soap were traced to the use of mowrah fat (I) (which contains a high proportion of unsaturated unsaponifiable constituents) in the stock, the oxidation being accelerated by traces of Cu derived, in one case, from the bronze of the stamping machines, and, in another, from Cu which is added to give a green colour to the partly-bleached (I) used by the soap-maker. E. L.

**Examination of fatty acids isolated from soaps.** G. WOLFF (Chim. et Ind., 1933, 29, Spec. No., 1039—1042).—M.p. (titer) curves for binary and ternary mixtures of the fatty acids from palm, coconut, arachis, linseed (I), and soya-bean (II) oils, and for tallow and horse grease, are plotted. Two anomalous small maxima in the curve for (I)-(II) fatty acids [at a point corresponding to 10% of (II)] seem indicative of sp. fatty acids. E. L.

**Working-up of soap stock from oil refining.** K. BANDAU (Fettchem. Umschau, 1933, 40, 157—159).—Various processes for the recovery of neutral fat from soap stock are discussed. E. L.

**Washing effect. II. Washing effect of the sodium salts of higher, homologous alkylsulphonic acids, compared with their foaming power, surface activity, and state of distribution in aqueous solution. III. Constitution and washing effect.** E. GÖTTE (Kolloid-Z., 1933, 64, 327—331, 331—335; cf. B., 1933, 797).—II. The detergent powers (I) of the Na salts of alkylsulphonic acids ( $C_{12}$ — $C_{18}$ ) have been investigated. A simple relation exists between the alkaline max. of the (I) and the chain-length of the mol. This max. is displaced in the direction of increasing chain-length by rise of temp. and by increase in the hardness of  $H_2O$ . In alkaline systems the (I) of the sulphonates examined is several times that of the soaps, whilst the difference is less marked in the neutral and acid regions. Foaming is most marked at concns. which lead to the formation of mol. aggregates, and bears no simple relation to (I).



III. The dirt present in soiled textiles is negatively charged. Detergents must therefore have surface-active anions, substances with surface-active cations being ineffective in spite of their high foaming power. (I) is least at the point of reversal of charge of the particles of dirt. The val. of a detergent can be estimated from the surface tension and foaming properties only in a homologous series.

E. S. H.

**Evaluation of foam-producing and wetting-out materials and detergents etc.** HETZER (Chem.-Ztg., 1933, 57, 715—716, 735—736).—The effects on the foam test of variations in experimental technique and in fatty acid content of the original oil are discussed. The foam val. (I) of soaps made from tallow are > those of soaps derived from palm-kernel oil or hardened oils, particularly if the air space in the shaking vol. is large, and for const. results this should be a min. (I) decreases with an increase in the age of the solution up to 12 hr., when it becomes const., this being partly accounted for by the longer duration of contact with air. To obtain const. results, 50 c.c. of fresh sample are shaken 40 times in a 120-c.c. cylinder at 20° and/or 50°, and the % of foam is read at 1-min. intervals over 10 min.

J. G.

**Oil recovery during the preheating [cooking] of the seeds (Skipin process).** R. HEUBLUM (Fettchem. Umschau, 1933, 40, 162—164).—Preliminary pressing may be omitted and a high proportion (40—60%) of the oil recovered directly in the kettle if the H<sub>2</sub>O content of the seed meal (sunflower, castor) is adjusted to 8.5% and the material is heated (in closed kettles) to 60°.

E. L.

**Determination of gossypol in crude cottonseed oil.** H. D. ROYCE (Oil and Soap, 1933, 10, 183—185).—In the pptn. of gossypol (I) by NH<sub>2</sub>Ph, excess of C<sub>5</sub>H<sub>5</sub>N (4:1) improves the yield of ppt. By this method (I) is shown to be present in crude hot-pressed oil. Samples of crude oils from 6 different mills gave 0.049—0.105% (I). The (I) in crude oils is rendered non-precipitable by heating the oil at 150° for 30 min.; at lower temp. the yield is decreased.

G. H. C.

**Gossypol in the technology of cottonseed oil.** H. D. ROYCE and F. A. LINDSEY, JUN. (Ind. Eng. Chem., 1933, 25, 1047—1050).—Gossypol (I) inhibits the oxidation of cottonseed oil (II) as measured by the time of fading of methylene-blue and by the peroxide-accelerated oxidation curve. (I) also inhibits the saponification of (II) by the Kontakt catalyst. The inhibition is > that of lecithin. (I) in hot-pressed (II) is detected by the NH<sub>2</sub>Ph—C<sub>5</sub>H<sub>5</sub>N method. Crude (II) may contain 0.2% of heat-modified (I) which is not precipitable by any method. Refining losses have been lowered 1—3% by the addition of <1% of pure (I).

E. C. S.

**Testing of stand oil.** H. WOLFF (Farbe u. Lack, 1933, 426).—Stand oils which have been over-polymerised undergo livering, e.g., with ZnO. To test such an oil benzene (I) is added; the highly polymerised portion is insol. The (I) used should have b.-p. range 80—150° and be freed from aromatic and naphthenic hydrocarbons by treatment with fuming H<sub>2</sub>SO<sub>4</sub>, washing, and drying with anhyd. Na<sub>2</sub>SO<sub>4</sub>.

S. M.

**Specifications for china-wood oil.** M. T. FRANÇOIS (Chim. et Ind., 1933, 29, Spec. No., 1051—1055).—Various official specifications are reviewed. Authentic samples of oil (extracted by light petroleum under CO<sub>2</sub>) from *Aleurites Fordii* (tung, 4 samples) and *A. montana* ("Abrasin," 2 samples) had, respectively: acid val. 0.53—0.84, 1.3—1.5;  $d_4^{20}$  0.9422—0.9489, 0.9340—0.9399;  $n_D^{20}$  1.51875—1.51992, 1.51012—1.51194. The  $n$  val. is a useful indication of purity, and the optical dispersion permits a distinction between the oils of *A. Fordii* and *A. montana*; the I val. is unsuitable as an analytical criterion.

E. L.

**Some lesser-known oils.** E. BUREŠ (Chim. et Ind., 1933, 29, Spec. No., 1056—1077).—Detailed analyses are given of the oils obtained by extraction (with a variety of solvents in each case) from the kernels of peach, apricot, and pear seeds, "Para" nuts, and seeds of *Raphanus raphanistrum*.

E. L.

**Determination of inflammability of textile oleines.** E. I. BETTER (Fettchem. Umschau, 1933, 40, 159—161).—Determination of the rate of increase of the peroxide val. (cf. B., 1931, 726) on exposure to light affords a sensitive measure of the oxidisability (inflammability) of oleines; the comparative results are in fair agreement with the conclusions reached from parallel tests by the Mackey method.

E. L.

**Train oil and its adulterants.** S. SALM (Allgem. Oel-u. Fett-Ind., 1933, 30, 385—387).—Several commercial mixtures labelled as "Kunsttran" or "Helltran" consist only of mineral oils mixed with > 10—20% of marine-animal oils.

E. L.

**Properties of halibut-liver oil.** R. T. M. HAINES and J. C. DRUMMOND (Brit. Med. J., 1933, i, 559—561).—There was no uniform correspondence between the unsaponifiable fraction (I) of halibut-liver oil and the vitamin-A content ("blue val.") although the oils with the greatest and least vitamin-A potency, respectively, had the highest and lowest (I). The oil with the highest (I) had the least cholesterol. The absorption at 328 mμ was proportional to the "blue vals." of the oils. Direct treatment of the halibut livers with steam did not break up the tissues as in the case of cods' livers.

NUTR. ABS. (B).

**Effect of the conditions of storage on the vitamin-D potency and other features of cod-liver oil.** E. J. SHEEY (Sci. Proc. Roy. Dublin Soc., 1933, 20, 463—468).—A good-quality cod-liver oil does not appreciably alter in vitamin-D potency when stored for 16 months.

H. G. R.

**Comparing the stability of [oil] emulsions with variation in the proportion of ingredients.** L. M. LANGEVIN (J. Amer. Pharm. Assoc., 1933, 22, 728—735).—Emulsions (5—55% of oil) of almond, castor, cod-liver, cottonseed, raw linseed, olive, and sesame oils, using acacia (I) as emulsifying agent (1:2 H<sub>2</sub>O:4 oil), may be prepared with large variations in the 1:2:4 proportions, which is not always the optimum. Increase in the proportion of (I) results in a decrease in size of the oil globules; increase in H<sub>2</sub>O proportion increases the size of the globules, and the more uniform is the size of the dispersed globules the more stable is



the emulsion. The smallest oil particles were present in emulsions containing 2—4 pts. of oil. E. H. S.

**Determination of the bromine index of oils.** R. MARCILLE (Ann. Falsif., 1933, 26, 393—398).—A slight excess of Br is added to a 3·3% solution of oil in Et<sub>2</sub>O and the ppt. is separated and washed with Et<sub>2</sub>O (all operations being carried out in a centrifuge tube) and dried at 110°; the Br index (I) is the wt. of ppt. per 100 g. of oil, and the error is  $\pm 2$  mg. Data given for a range of mixtures containing linseed and various fish oils show that the wt. of Br ppt. is not proportional to the amount of pptg. oil present. Pure commercial linseed oil has (I) > 45; (I) increases on ageing, especially with fish oils. The presence of AcOH gives unsatisfactory results (cf. following abstract).

J. G.

**Analysis of fish-preserving oils.** R. MARCILLE (Ann. Falsif., 1933, 26, 398—403).—By the method of mixtures it is possible to determine the amounts of fish oil present in admixture with olive oil from the *n*, Br ppt. (cf. preceding abstract), and I val. Allowance must be made for the presence of any other vegetable oils and, since young sardines have a lower *n*, for the size of the fish; appropriate data for the pure oils are given. It is shown that *n* of the vegetable oil may then be calc. from the above data, and an indication obtained as to its nature if other than olive oil. Satisfactory results were obtained with sardine, sprat, and tunny oils, but cod-liver oil gave a Br ppt. which was collected with difficulty.

J. G.

**Portuguese waxes.** C. LEPIERRE and A. DE CARVALHO (Chim. et Ind., 1933, 29, Spec. No., 1087—1093).—Mean, max., and min. vals. are given for the analytical consts. of yellow beeswax from Portugal and Angola and of white beeswax from Portugal. The means are: acid val. 19, 19·5, 19·5; ester val. 72·5, 73, 73; sap. val. 91·5, 92·5, 92·5; I val. 10·5—11, 12—12·5, 6; Hübl's ratio (combined acid/free acid) 3·85, 3·75, 3·75.

A. G.

**Erratum.**—On p. 797, col. 2, line 23 from the bottom, for GOTTE read GÖTTE.

**Hydrogenation.**—See I. Corrosion in sulphonators.—See X. Ageing of cresol soap solution.—See XX.

See also A., Oct., 1933, Prep. of pure sulphuric esters of hydroxystearic acid. 1034, Japanese beeswax. 1035, Vitamins (various). 1066, Insect wax.

## PATENTS.

**Inhibiting oxidation of unsaturated fats, oils, fatty acids, and substances containing fatty material having a tendency to become rancid.** G. R. GREENBANK, Dedicator to GOVT. AND PEOPLE OF THE UNITED STATES (U.S.P. 1,898,363, 21.2.33. Appl., 16.9.32).—Unsaturated, polybasic aliphatic acids of the type of maleic, fumaric, aconitic, citraconic, and itaconic acids, or their esters or anhydrides, are added to inhibit rancidity in edible fatty products.

E. L.

**Production of a soap product.** D. R. LAMONT, ASSR. to COLGATE-PALMOLIVE-PEET Co. and PROCTER & GAMBLE Co. (U.S.P. 1,897,701, 14.2.33. Appl.,

23.4.31).—A readily-sol. soap powder is prepared by incorporating a gas (air etc.) under pressure in the liquid soap, which is then sprayed into a region of reduced pressure, in order to expand the dissolved gas and impart a cellular structure to the soap particles.

E. L.

**Manufacture of soap intended more particularly for preparation of solid or consistent grease.** C. DEGUIDE (B.P. 398,402, 14.2.33. Fr., 15.2.32).—Fats are saponified with cryst. Ba(OH)<sub>2</sub>, and the soap is emulsified with mineral oil.

E. L.

**Mixed acid anhydrides. Wetting etc. agents.**—See III.

## XIII.—PAINTS; PIGMENTS; VARNISHES; RESINS.

**Metallic driers.** PHILADELPHIA CLUB (Amer. Paint & Varnish Manufs.' Assoc., Circ. 423, Nov., 1932, 12 pp.).—The effects of driers on the induction period, gain in wt., and chemical changes occurring in the drying of oil films were studied. The type of chemical combination of the metal in the drier was also considered. Driers shorten the induction period by counteracting the anti-oxidants present in the oil. Driers accelerate the rate of oxidation, formation of peroxides, saturation of double linkings, and gelation process of oil. The drying action for a given % of metal depends on the degree of dispersion and "availability" of the metal rather than on any sp. effect of the org. radical with which it is combined.

S. S. W.

**Effects of driers on durability.** CINCINNATI-DAYTON-INDIANAPOLIS CLUB (Amer. Paint & Varnish Manufs.' Assoc., Circ. 423, Nov., 1932, 4 pp.).—A preliminary report is given on a series of exposures of "single-drier" linseed and tung oil varnishes over primed and unprimed steel. Each drier was added as linoleate and as naphthenate. Starting with 0·038% Co on the wt. of oil, the individual amounts of driers used were Co:Mn:Pb::1:1:4 and increasing amounts of drier in the ratio 1:3:7:10. The drying times of all the varnishes are recorded. After 11 weeks all were giving satisfactory protection and no definite comparisons could be made.

S. S. W.

**Effect of synthetic resins and pigments on drier absorption and viscosity.** CALIFORNIA CLUB (Amer. Paint & Varnish Manufs.' Assoc., Circ. 423, Nov., 1932, 5 pp.).—The viscosity, drying time, and degree of settling of a range of paints comprising typical pigments and synthetic resin varnishes, and the viscosities of the varnishes themselves, were observed over 7 months. The conclusions reached are: viscosity of correctly cooked synthetic resin varnishes is reasonably const.; synthetic resins do not absorb driers; pigments may have fattening effect on these varnishes (lithopone, chrome yellow, Venetian red, Prussian blue), and may absorb driers (C black, Prussian blue, toluidine toner, Ti pigments).

S. S. W.

**Glycerol phthalate solid colours.** H. A. GARDNER and L. P. HART (Amer. Paint & Varnish Manufs.' Assoc., Circ. 420, Oct., 1932, 278—280).—Chrome yellow, chrome green, and toluidine red were incorporated with varnishes made from 5 alkyd resins (I),



a phenolic resin (II), and bodied tung oil varnish, and the enamels exposed on wood and metal. Little breakdown had occurred after 16 weeks. (II) "chalks" and, when clear, yellows more readily than (I), among which "alkyd BR" was least satisfactory. S. M.

**Apparatus for testing the mechanical strength of paint and lacquer films, and of plastic masses.** K. E. KRAUSE and N. V. SMIRNOV (J. Appl. Chem. Russ., 1933, 6, 749—752).—Apparatus is described for determining tensile strength and plasticity. R. T.

**Influence of various ingredients on the yellowing of films.** A. W. VAN HEUCKEROTH (Amer. Paint & Varnish Manufs.' Assoc., Circ. 415, Oct., 1932, 239—250).—The yellowing of nitrocellulose-synthetic resin and oil films under humid conditions in the dark and under the action of artificial light was studied with reference to variations in resin, plasticiser, drier, and pigment combination. It was found that yellowing results obtained by exposure to the light from an incandescence lamp and that from a quartz-Hg-vapour lamp are not necessarily of the same order. The degrees of yellowing with various combinations of ingredients and testing conditions are summarised graphically. S. S. W.

**Relative merits of spray and brush painting.** CHICAGO CLUB (Amer. Paint & Varnish Manufs.' Assoc., Circ. 423, Nov., 1932, 4 pp.).—Brush and spray trials of a linseed oil paint compared with 3 synthetic resin paints over a special primer (applied at two consistencies each by brush and spray) on various types of wood have been inaugurated. A preliminary report of the condition of the panels after 6½ months' exposure is given. No marked failure occurred, and significant comparison of brush and spray methods could not be made. S. S. W.

**Relationship between radiant energy and paint.** F. C. ARWOOD (Amer. Paint & Varnish Manufs.' Assoc., Circ. 423, Nov., 1932, 8 pp.).—A general summary is given of the principles of radiation and the thermal insulation properties of paints of various types. Paints that reflect as well in the infra-red region as do white paints in the visible spectrum are not available. Details of the heat losses of building boards and thermometer bulbs coated in various ways are given. S. S. W.

**Effect of china-wood [tung] and fish oils in outside white paints.** TORONTO CLUB (Amer. Paint & Varnish Manufs.' Assoc., Circ. 423, Nov., 1932, 1 p.).—A preliminary account is given of exposures of straight linseed oil paints and similar paints in which fish oil and tung oil have been incorporated. S. S. W.

**Causes of and remedies for paint failure on plaster surfaces.** MONTREAL CLUB (Amer. Paint & Varnish Manufs.' Assoc., Circ. 423, Nov., 1932, 15 pp.).—Current plastering procedure is summarised and a comprehensive series of tests is described, covering the use of a standard blue-tinted long-oil varnish paint over the various known types of sealers, each on panels representing good and faulty plastering. The latter include soft panels for excessive suction, hard panels for shrinkage cracks and hard spots, frozen panels, efflorescent panels, and panels containing free CaO. The behaviour of the panels on normal exposure and on semi-immersion in

H<sub>2</sub>O for periods up to 1080 hr. are tabulated. The general conclusion is that paint failures on plaster are almost invariably due to faulty plastering, insufficient ventilation, etc. S. S. W.

(A) Paints for exhibit buildings. (B) Adhesion of coatings to Parkerised and Bonderised metal. (C) Preservation of fresco for exterior decoration. H. A. GARDNER and G. G. SWARD (Amer. Paint & Varnish Manufs.' Assoc., Oct., 1932, Circ. 421, 281—288).—(A) Casein paints are not suitable for Douglas fir plywood and "sheet rock"; 4 satisfactory mixtures are given. (B) Some exposure tests indicated that Parkerised surfaces were better preserved than Bonderised surfaces. (C) Of 8 various compositions a synthetic resin-tung oil varnish provided the best protection. Pigmented Si ester paint should also be suitable. S. M.

**Built-up coatings for underground pipes [in hot areas].** H. A. GARDNER and L. P. HART (Amer. Paint & Varnish Manufs.' Assoc., Oct., 1932, Circ. 418, 270—273).—Coatings of widely varying composition were tested by burying the covered pipes for 4 months in cinders and S which were wetted daily with 0.1% aq. H<sub>2</sub>SO<sub>4</sub>. Satisfactory protection was obtained with spiral wrappings of flexible celluloid in varnish applied on a dense ZnCrO<sub>4</sub> primer and covered with rubber-impregnated cotton tape. S. M.

(A) Painting sheet copper. (B) Copper and other metallic powders in paints. (C) Copper stains on white paint. H. A. GARDNER and L. P. HART (Amer. Paint & Varnish Manufs.' Assoc., Oct., 1932, Circ. 416, 251—262).—(A, C) Linseed oil paints and long oil enamels adhere satisfactorily to unroughened Cu sheets, which should be primed with a varnish or enamel to prevent staining of the finishing coat. (B) Paints made from Cu powder and linseed oil or asphalt afford only temporary protection to Fe; rapid corrosion ensues when Cu powder is dusted upon a tacky, varnished surface. Al and Zn powder are less active. S. M.

**Accelerated tests for metal preservatives for sub-sea-water service.** H. A. GARDNER and G. G. SWARD (Amer. Paint & Varnish Manufs.' Assoc., Oct., 1932, Circ. 417, 263—269).—The protection afforded against the rusting of steel by 8 linseed oil paints (I) and by Al bronze in a spar varnish (II) was tested by exposure at 45° inclination during the day and immersion in 3% NaCl solution during the night for 6 weeks. Results are photographed. Addition of an alkyd resin to (I), with consequent reduction of the pigment concn., was without advantage except in the case of the basic Pb chromate paints; graphite paints were useless. (II) was effective only if 2 coats were applied. S. M.

**Industrial microscopy. II. Paint pigments.** C. H. BUTCHER (Ind. Chem., 1933, 9, 351—352).—A review of methods.

**Preparation of chrome-yellow from lead formate.** N. N. EFREMOV and A. A. VESELOVSKI (J. Appl. Chem. Russ., 1933, 6, 665—668).—Good-quality chrome-yellow is obtained by adding K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> and H<sub>2</sub>SO<sub>4</sub> to saturated aq. (HCO<sub>2</sub>)<sub>2</sub>Pb, or to its paste with an equal wt. of H<sub>2</sub>O. R. T.



**Darkening of chromium oxide green at high temperatures.** L. VANINO (Farben-Chem., 1933, 4, 327—328).—Both the matt and the fiery commercial  $\text{Cr}_2\text{O}_3$  greens rapidly become greyish-black or black when strongly heated. The change is attributed to the formation of a higher oxide, and is not due to the presence of org. matter or Cu. S. M.

**RAL-method for analysis of red lead.** J. F. SACHER (Farbe u. Lack, 1933, 403—404).—The RAL (Reichsausschuss f. Lieferbedingungen) method for determining  $\text{PbSO}_4$  and Pb in the insol. residue obtained after treating the sample of  $\text{Pb}_3\text{O}_4$  with  $\text{HNO}_3$  and  $\text{H}_2\text{O}_2$  is recommended. In the determination of the  $\text{PbO}_2$  content (B., 1911, 802) considerable excess of  $\text{H}_2\text{C}_2\text{O}_4$  leads to high results (cf. B., 1930, 917). S. M.

**Heavy and light zinc white.** M. MOLDENHAUER (Farben-Chem., 1933, 4, 165—168).—The manufacture of ZnO pigment is outlined and a discussion is given of the effects of velocity of gas stream, quantity of air supplied, duration of heating, and variation in the plant details on the apparent density ( $D$ ) of the product.  $D$  increases frequently when the Zn white is packed and stored, but can be reduced to 0.4—0.5 kg./litre by sieving. Wet pptn. *e.g.*, of basic  $\text{ZnCO}_3$ , followed by calcination yields a lighter product. S. M.

**Cadmium, thallium, indium, and gallium as by-products of the lithopone industry.** W. N. HIRSCHER (Chem. and Ind., 1933, 797—798).—Zn solutions for the prep. of lithopone must be freed from these metals, and the residues contain them in valuable amounts. The Cd is pptd. by means of Zn fume ("blue powder") and the Tl as  $\text{TlCl}$ . G. H. C.

**Determination of degree of dispersion of pigments and finely-divided substances by mechanical and optical methods.** F. SCHMID (Chem. Fabr., 1933, 6, 349—352).—The methods of dry- (I) and wet-rubbing (II) between the fingers, sieving (III), sedimentation (IV), and microscopical examination are reviewed. Using (I), particles  $> 100 \mu$  diam. impart grittiness, whilst material which consists of particles  $< 10 \mu$  diam. is floury; (II) is more sensitive than (I). (III) serves only for the separation and determination of the coarse content; (IV) is useful for particles down to  $5 \mu$  diam. The technique of (V) is discussed; with diffused illumination in ultra-violet fluorescent light it is possible to detect and observe the geometrical structure of particles having diam. as low as  $0.1 \mu$ . Photomicrographs are given of 6 different samples of Zn white and of very fine bronze wire and silk sieves after considerable use. S. M.

**Standards for fineness of pigment grinding.** NEW YORK CLUB (Amer. Paint & Varnish Manufrs.' Assoc., Circ. 423, Nov., 1932, 3 pp.).—The factors affecting pigment grinding are summarised and the need for comparative standards is stressed. A series of pastes were prepared incorporating (by stirring) Aloxite of different grades of coarseness with a finely-ground White Seal ZnO-alkali refined linseed oil paste. It is shown that the use of such standards improves uniformity in practice. S. S. W.

**Coloured waterproof drawing inks.** E. W. ZIMMERMAN (Ind. Eng. Chem., 1933, 25, 1033—1034).—

Such inks comprise 5% solutions of shellac (in dil. aq.  $\text{NH}_3$ , containing a little  $\text{Na}_2\text{B}_4\text{O}_7$  as insolubiliser) and suitable dyes. Unbleached shellac, freed from insol. waxes, orpiment, and dirt, was found satisfactory. Out of 92 dyes examined, the following were found not to change colour or deteriorate after storage for 2 years: Erythrosine Yellow, Brilliant Orange R, Chloramine Yellow, Brilliant Milling Green B, Wool Blue G extra, Me-Violet B, and Benzamine Brown 3GO. Methods of testing  $\text{H}_2\text{O}$ -fast inks are detailed. S. S. W.

**Blue dye as evidence of age of writing.** C. E. WATERS (Ind. Eng. Chem., 1933, 25, 1034—1035).—Previous statements that blue dye (I) in writing done with ordinary blue-black ink is completely oxidised away in 15 years are disproved. Of 147 documents examined, which were  $> 15$  (and some  $> 50$ ) years old, 78 showed the presence of (I) when tested with distilled  $\text{H}_2\text{O}$ . The presence or absence of dye cannot be taken as evidence of the age of writing. S. S. W.

**Soaps. IV. Effect of metallic resins in varnish.** PHILADELPHIA CLUB (Amer. Paint & Varnish Manufrs.' Assoc., Nov., 1932, Circ. 423, 3 pp.).—The viscosity, colour, drying time, degree of skinning in the can, kauri reduction test,  $\text{H}_2\text{O}$ -resistance (hot and cold), and durability of varnishes containing equiv. amounts of metal resins as single driers were observed. The driers used were pptd. Ca, Pb, Al, Mg, and Zn resins of metal content 6.4, 25.2, 3.8, 2.9, and 9.6% respectively, and fused Na resinate, together with a mixed Pb-Co-Mn resinate for comparison. The results are discussed but no general conclusions can be drawn. S. S. W.

**Possible effect of anti-skinning agents in increasing the life of varnish.** H. A. GARDNER and J. R. STEWART (Amer. Paint & Varnish Manufrs.' Assoc., Oct., 1932, Circ. 419, 274—277).—Addition of 0.72% (calc. on varnish base) of 13 anti-oxidants to modified phenol resin-tung oil varnishes usually increased the resistance to exposure (I), but with 0.072% (I) usually decreased. The anti-skinning effect and the influence on (I) were not parallel. Partial substitution of mineral spirits, used as thinner, by *o*- $\text{C}_6\text{H}_4\text{Cl}_2$ , wood turpentine, dipentene, or a ketone-terpene hydrocarbon also increased (I) (cf. B., 1933, 77). S. M.

**[Tung oil] varnishes produced by blowing.** H. A. GARDNER (Amer. Paint & Varnish Manufrs.' Assoc., Sept., 1932, Circ. 414, 223—238).—Blowing air through tung oil at room temp. or, better, at  $100^\circ$  tends to inhibit the "frosting." By incorporating resins (*e.g.*, ester gums, modified  $\text{PhOH-CH}_2\text{O}$  resins), solvents, preferably turpentine, and driers with the oil before blowing, "gas-proof" low-cooked tung oil varnishes can be obtained. Some general notes on the blowing procedure and the properties imparted, as well as preliminary (5 months') exposure results of varnishes so made, are given. S. S. W.

**Flexible alkyd resins.** R. H. KIENLE and P. F. SCHLINGMAN (Ind. Eng. Chem., 1933, 25, 971—975).—From the theory of synthetic resin formation it is considered that if long-chain heat-non-convertible mols. (I), *e.g.*, dihydric alcohol-dibasic aliphatic esters such as



glycol succinate, were attached through primary-valency linkings to rigid heat-convertible mols., *e.g.*, glyceryl phthalate, flexible heat-convertible resins should result. This received confirmation practically, the flexibility and plasticity being controllable by increase in the proportion of (I). Compositions and properties of such combinations are detailed and their industrial possibilities as film-forming materials, celluloid substitutes, artificial rubber, etc. are outlined.

S. S. W.

**Resinification of glycerol.** S. N. USHAKOV and E. M. OBRIADINA (Ind. Eng. Chem., 1933, **25**, 997—1001).—The optimum conditions for formation of aldehydic resins by heating glycerol with a catalyst were studied, *e.g.*, nature and amount of catalyst, reaction period, etc.  $\text{H}_2\text{SO}_3$  and  $\text{H}_2\text{SO}_4$  and their easily reducible salts are suitable catalysts, best results being given by  $\text{Hg}_2\text{SO}_4$  and  $\text{CuSO}_4$ . Dark-coloured viscous resins sol. in  $\text{H}_2\text{O}$ , EtOH, EtOH- $\text{C}_6\text{H}_6$ , and  $\text{COMe}_2$  (partly) result. On heating in thin layers, glossy flexible films insol. in  $\text{H}_2\text{O}$ , EtOH, and  $\text{COMe}_2$ , and unaffected by weak acids and alkalis, are obtained. The uses of these resins to impart elasticity to  $\text{PhOH-CH}_2\text{O}$  products of "resol" or "novolak" type without affecting their resistant properties, and as plasticisers for albuminous plastics, are indicated.

S. S. W.

**Causes of the dark colour assumed by "novolak" phenol-aldehyde resins.** B. B. MASHKILLEISON and S. S. ZIMMERMAN (Plast. Massi, 1932, No. 1, 32—37).—The nature of the catalyst influences the colour and its stability. Max. colour stability is attained by the use of weak mineral acids, particularly  $\text{H}_3\text{PO}_4$ . Metallic salts of mineral acids are least satisfactory. The nature of the catalyst also affects m.p., solubility, and turbidity point in presence of rosin.

CH. ABS.

**Petroleum solvents.**—See II. Camphene from turpentine.—See III. Leather cloth. Cellulose derivatives.—See V. Behaviour of rust-protecting coatings.—See X. Testing stand oil.—See XII. Pb poisoning from paints.—See XXIII.

See also A., Oct., 1027, Egyptian pigments. 1046, Condensation of  $\text{CH}_2\text{O}$ , amines, and phenols.

## PATENTS.

**Production of white lead.** J. W. LIFF and W. J. LINDSAY, Assrs. to NAT. LEAD CO. (U.S.P. 1,898,054, 21.2.33. Appl., 10.8.26. Renewed 20.2.29).—Granulated Pb is agitated with AcOH and  $\text{H}_2\text{O}$  in a rotating drum through which a current of air and  $\text{CO}_2$  is passed, and the charge is subjected to frequent, brief, and substantially regular moistening and drying cycles, the  $\text{H}_2\text{O}$  content being maintained between suitable limits. > 90% conversion is achieved in 3 days.

S. S. W.

**[Ink for] protection and authentication of articles [cheques etc.].** E. BAYLE, H. GEORGE, and A. MACHÉ (U.S.P. 1,899,452, 28.2.33. Appl., 24.1.29. Fr., 28.1.28).—An ink is used the colour of which is a function of  $p_{\text{H}}$ . It is a mixture of the  $\text{ZnCl}_2$  salts of hexamethyl- and pentamethylmonoethyl-*p*-rosaniline, the disazo dye from tetrazotised *o*-tolidine and 1:4- $\text{C}_{10}\text{H}_6(\text{NH}_2)_2\text{SO}_3\text{Na}$ , NaOH, and  $\text{NaH}_2\text{PO}_4$ . H. A. P.

**Refining of rosin.** I. W. HUMPHREY, Assr. to HERCULES POWDER CO. (U.S.P. 1,897,379, 14.2.33. Appl., 11.8.28). A solution of rosin (I) in gasoline is agitated with large proportions of MeOH, which preferably contains 2—8% of  $\text{H}_2\text{O}$ , cooled to below  $0^\circ$ , and the decolorised (I) recovered from the gasoline layer. Alternatively, (I) is distilled and recrystallised from MeOH (cf. B., 1933, 756).

S. M.

**Dental plate base.** J. F. WALSH, Assr. to CELLULOSE CORP. (U.S.P. 1,897,286, 14.2.33. Appl., 1.12.28).—The use of cellulose nitrate plasticised with Bu<sub>2</sub> phthalate is claimed.

S. M.

**Manufacture of thermoplastic resinous materials.** IMPERIAL CHEM. INDUSTRIES, LTD., and A. RENFREW (B.P. 398,194, 29.2.32).—To remove gas bubbles from compact masses of vinyl acetate and other resins, the material is covered with  $\text{H}_2\text{O}$ , liquid paraffin, or other inert liquid in an open container which is heated (autoclave) above the softening point of the resin and under 2—50 atm. pressure.

S. M.

(A) Artificial resin from glycerol and the like. (B) [Urea-aldehyde] resinous condensation product. C. ELLIS, Assr. to ELLIS-FOSTER CO. (U.S.P. 1,897,977—8, 14.2.33. Appl., [A] 26.12.22, [B] 1.12.28).—(A) In preparing glyptal resins the solidification temp. is lowered by adding CaO or other alkali,  $(\text{NH}_4)_2\text{S}_2\text{O}_8$ , or a multivalent metal compound which can combine with the free acids formed. (B) The acid reaction product of urea and  $\text{CH}_2\text{O}$ , or derivatives of these compounds (cf. U.S.P. 1,846,853; B., 1932, 1091), is incorporated with either a  $\text{CS}(\text{NH}_2)_2\text{-CH}_2\text{O}$  resin, a  $\text{PhOH-S}$  resin (prepared by the action of  $\text{S}_2\text{Cl}_2$  on  $\text{PhOH}$ ), or both, in sufficient quantity to give a readily fusible material when heated under pressure.

S. M.

**Softeners. Plasticisers. Condensation products.**—See III. Pigments.—See III and IV. Stencil and parchment papers.—See V.

## XIV.—INDIA-RUBBER; GUTTA-PERCHA.

**Dispensibility of gas black [in rubber]. I. Methods of measuring gas-black dispersion.** R. P. ALLEN and F. K. SCHOENFELD (Ind. Eng. Chem., 1933, **25**, 994—997).—The different methods of preparing a section of a rubber block for visual or microscopical observation are considered. Examination of a torn surface is preferred, and the specimen is compared with a series of "standardised" sections.

C. A. K.

**Chemistry of soft rubber vulcanisation. I. Measurement of vulcanisation.** B. S. GARVEY, JUN., and W. D. WHITE (Ind. Eng. Chem., 1933, **25**, 1042—1046).—Tests indicative of the changes occurring during vulcanisation include rate and degree of freezing of a stretched sample in  $\text{H}_2\text{O}$  at  $0^\circ$ , behaviour under tension in  $\text{H}_2\text{O}$  at  $90^\circ$ , behaviour on milling or when immersed in  $\text{C}_6\text{H}_6$ , plasticity (I) and its variation with temp., relation of retentivity to softness, hysteresis (II), stress-strain behaviour (III), and combined S. No single test is adequate as a measure of the degree of vulcanisation, but for stocks containing a high proportion of rubber a satisfactory group of tests includes (I), (II), and (III). The various properties do not alter at the same rate with increase in the degree of vulcanisation.

D. F. T.



**Reactions during vulcanisation [of rubber]. III. Multiple-accelerator effect.** H. C. JONES (Ind. Eng. Chem., 1933, 25, 1009–1011; cf. B., 1932, 651).—In a vulcanisable rubber mixing deficient in fatty acids, diphenylguanidine (I) activates mercaptobenzthiazole (II) by rendering the ZnO sol. and effective. In the presence of added fatty acids, (I) itself also acts as an accelerator so that the mixing vulcanises more rapidly than it would do with either (I) or (II) alone. But-aldehydeaniline behaves similarly to (I). Experiments in a mixture of  $C_6H_6$  and MeOH saturated with  $H_2S$  indicate that fatty acids increase the solubility of ZnO in the presence of (II), but reduce it in the presence of (I). These results are in accord with the improvement in tensile properties of a stock containing (II) and their impairment in a stock containing (I), on the addition of 3% of stearic acid. D. F. T.

**Notch- and tearing-strength of anisotropic rubber sheets.** F. KIRCHHOFF (Kautschuk, 1933, 9, 130–134).—Evaporation of mixtures of centrifugally conc. latex with 20% of shellac, the latter being introduced as its  $NH_4$  soap, gave films (1 mm. thick) which after being notched offered considerable resistance to tearing, the course of the tear being sickle-shaped. If, however, the films had been stretched several times to 100–200% of their original length and relaxed, they tore easily and straight in the direction of the earlier extension; this characteristic persisted even after a short time at 80°, but across the direction of extension the tear was sickle-shaped. In the absence of shellac, latex films which had been stretched and then heated in a relaxed condition showed no tendency to preferential tearing in the direction of the earlier extension. The shellac consequently must have led to a partial fixation of the deformation of the rubber particles, leading to "grain" similar to that produced by calendaring; the persistence of anisotropy was also revealed by the Röntgen diagram. D. F. T.

**Effect of temperature and frequency on the dielectric constant, power factor, and conductivity of compounds of purified rubber and sulphur.** A. H. SCOTT, A. T. MCPHERSON, and H. L. CURTIS (Bur. Stand. J. Res., 1933, 11, 173–209).—At 25° and 1000 cycles, the dielectric const. of rubber was increased by added S to a max. (3.75) with 11.5% S; it then decreased to a min. (2.70) with 22% S, and increased with further additions. The power factor varied similarly. Vals. for frequencies between 60 and 300,000 cycles and from –75° to 235° are tabulated. H. J. E.

**Testing of the tinning of rubber-insulated conductors.** A. R. MATTHIS (Chim. et Ind., 1933, 29, Spec. No., 235–244; cf. B., 1931, 452).—A definite surface of the tinned wire is placed in contact with aq.  $NH_3$  ( $d_0$  0.910) in the presence of air for 5 min. The  $NH_3$  attacks the badly tinned portions, forming  $Cu(NH_3)_4(NO_2)_2$ . The Cu dissolved is determined electrolytically or colorimetrically. The results of a large no. of tests are tabulated. J. W. S.

**New technique in the rubber industry. [Manufacture of "Filastic."]** J. C. BONGRAND (Chim. et Ind., 1933, 29, Spec. No., 1080–1084).—Photomicrographs illustrate how "filastic" (rubberised cotton etc.) threads (F) are uniformly impregnated throughout

the fibre with non-masticated virgin rubber. F without further coating adhere well to each other or to masticated rubber during vulcanisation. Uses of F and of the highly durable woven and vulcanised fabrics made therefrom are indicated. E. L.

**Flexible resins.**—See XIII.

See also A., Oct., 1054, **Polymerisation of caoutchouc, isoprene, and styrene.**

#### PATENTS.

**Compounding of rubber.** W. S. CALCOTT, W. A. DOUGLASS, and O. M. HAYDEN, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,899,554, 28.2.33. Appl., 13.12.27).—The uniform incorporation of compounding ingredients is effected by first reducing them to colloidal dimensions in the presence of a dispersing agent which is itself sol. in rubber. A colloidal dispersion of  $\alpha$ - $C_{10}H_7$ -NHPh and  $m$ - $C_6H_3Me(NH_2)_2$  in stearic acid is instanced. D. F. T.

**Processing and compounding of rubber.** H. G. C. FAIRWEATHER. From R. J. KING CO., INC. (B.P. 398,702, 9.1.33).—The milling and mixing operations are shortened by adding to the unplasticised rubber a thermoplasticiser comprising a solution of "solvent activators" in a solvent such as "mineral seal oil," which is also a solvent for rubber. The solvent activators, which promote the softening action of the solvent on the rubber, may consist of oil-sol. sulphonic acids of the paraffin or aromatic series and alcohols  $> C_3$ . D. F. T.

**Manufacture of rubber products.** M. DUPRET (B.P. 398,780, 16.5.33. Addn. to B.P. 396,503; B., 1933, 880).—Any aq. dispersion of rubber may be used instead of latex, and the reinforcing material may be covered with soft rubber on both sides. D. F. T.

**Dispersion of rubber in water.** E. B. NEWTON, Assr. to B. F. GOODRICH CO. (U.S.P. 1,899,243, 28.2.33. Appl., 3.12.29).—A  $H_2O$ -insol. soap, e.g., Ca oleate,  $H_2O$ , and a peptising agent such as K oleate are successively incorporated in the rubber; the last-named addition effects inversion of the phases. A hydrophilic colloid, e.g., gum arabic, may be introduced with the  $H_2O$ . D. F. T.

**Production of [sprayed] rubber coverings.** INTERNAT. GEN. ELECTRIC CO., INC., Asses. of ALLGEM. ELEKTRICITÄTS-GES. (B.P. 398,477, 10.3.32. Ger., 10.3.31).—For spraying purposes solutions of rubber of substantially the viscosity of  $H_2O$  are used, to which may be added protective colloids such as soaps or oils for prevention of clogging of the atomiser nozzle. [Stat. ref.] D. F. T.

**Rubber composition and method of preserving rubber.** M. C. REED, Assr. to B. F. GOODRICH CO. (U.S.P. 1,899,058, 28.2.33. Appl., 7.10.29).—A compound of the structure  $\begin{matrix} C' \\ | \\ C'' \end{matrix} > N \cdot R \cdot X$ , where  $C'$  and  $C''$  are terminal C atoms of a chain, R is an aromatic nucleus, and X is H, OH, NO, or  $NH_2$ , e.g.,  $p$ -hydroxy-N-phenylmorpholine, is used as an antioxidant. D. F. T.



**Rubber composition and method of preserving rubber.** A. W. SLOAN, Assr. to B. F. GOODRICH CO. (U.S.P. 1,899,120, 28.2.33. Appl., 27.3.31).—*tert*-Aminophenols, especially the dialkylamino-compounds, e.g., dimethyl- and diethyl-*m*-aminophenol, are good antioxidants. D. F. T.

**Vulcanisation accelerators.**—See III.

#### XV.—LEATHER; GLUE.

**Evaluation of bating materials [for hides and skins].** K. H. GOELLER (Collegium, 1933, 547—551; cf. B., 1933, 277).—All casein-substrate methods of evaluation of bating materials are subject to an  $\text{NH}_4$  salt error. A method free from such error is described in which a weighed quantity of hide powder (*A*) is treated with the bating material (*B*) under standard conditions; after a definite period the residual powder is filtered, washed, and dried. The amount of *A* dissolved is a measure of the activity of *B*. D. W.

**Extraction of raw tanning materials [for analysis].** Committee report [of the American Leather Chemists' Association], 1933. C. A. BLAIR (J. Amer. Leather Chem. Assoc., 1933, 28, 423—432).—Boiling  $\text{H}_2\text{O}$  can be used instead of cold  $\text{H}_2\text{O}$  for digesting the samples. 0.60% more tannin was extracted in 7 hr. than in 4 hr. and 0.66% more tannin by sampling valonia unground instead of ground. The tannin was more easily and uniformly extracted than were the non-tans and insol. matter. The adoption of a standard type of extractor is advocated. D. W.

**Determination of insoluble matter [in tannin analysis].** A.L.C.A. [American Leather Chemists' Association] Committee report, 1933. J. S. ROGERS (J. Amer. Leather Chem. Assoc., 1933, 28, 432—460).—1% more insol. matter was obtained by a modified Riess method (I) as compared with the official American method (II). The adsorption of tannin by the filter paper in method (II) was unaffected by tanning the paper. Concordant results were obtained by method (I), but it is slow with some extracts. D. W.

**Evaluation of tanning materials for tanning fishing nets.** J. OLIE and G. BROUWER (Collegium, 1933, 541—547).—The tannin content, total matter absorbed (*A*) by cotton from a 2% solution, the residual matter (*B*) on the treated cotton after washing for 16 hr., and the ratio *B/A* were determined for different samples of cutch (catechu), gambier, and wattle-bark extract (I); the highest ratio was given by (I). D. W.

**Tanning effect of sulphite-cellulose [waste] extracts.** W. VOGEL (Collegium, 1933, 524—531).—The use of 20—25% of sulphite-cellulose waste extract (I) is recommended in mixtures of tanning extracts for finishing off sole leather. It is not recommended to be used alone, but should always be mixed with some of the other tanning extracts. Analyses of leathers tanned with these different products are given to prove that (I) has definite tanning properties. D. W.

**Tan liquor heating device.** J. STARLING (Collegium, 1933, 538—540).—A portable cylinder (*A*) (20 cm. diam.) contains a steam heating coil and a small motor-driven propeller for circulating tan liquors through *A*. D. W.

**Determination of acid in vegetable tanned leather.** Report of [I.V.L.I.C.] Commission on leather analysis. L. JABLONSKI (Collegium, 1933, 523—524).—The Innes method is proposed as the official method and the  $p_{\text{H}}$  vals. should be determined by the quinhydrone in preference to the colorimetric method. The limiting vals. proposed by Innes (B., 1931, 938) and Kubelka (B., 1931, 819, 1064) should be used. D. W.

**Determination of nitrogen in leather.** C. VAN DER HOEVEN (Collegium, 1933, 532—538).—The ter Meulen-Heslinga method is used, in which the leather is heated with finely-divided Ni to 250° in a current of  $\text{H}_2$ ; the  $\text{NH}_3$  formed is collected and titrated. The method combines speed with accuracy. D. W.

**Defects in dried [raw] goatskins.** E. BELAVSKY (Collegium, 1933, 551—555).—The fineness of the grain is affected by the age of the animal and the climate. Animals in tropical countries have short hair and a fine-grained skin; those in colder regions possess longer hair and a coarser-textured skin. The grain tends to deteriorate as the skins become larger. The grease content of raw goatskins varies up to 24%, but a portion of this is removed in the manufacturing processes. Fat stains in finished goat leathers are attributed to the irregular distribution of the grease in the raw skins. Those parts which contain an abnormal amount of grease retain some after bating, and the amount is increased by fat-liquoring and drying. White specks may appear on storage. Some other stains on Indian dyed goat-skins have been traced to the tanning properties of the Indian "Khari" salt, which thereby prevents the affected places from swelling and tanning. The stains were not observed on "flint-dried" skins from the same province. D. W.

**Fat distribution in the bend portion of greasy hides and leather.** I. D. CLARKE and R. W. FREY (J. Amer. Leather Chem. Assoc., 1933, 28, 416—423).—The fat content (*F*) of samples taken from different parts of greasy and non-greasy raw hides and from fat-stained tanned bends was a max. in the kidney region, a min. in the corresponding region on the belly edge, and low at the shoulder end. *F* varied considerably in greasy hides and bends. Very little added oil is taken up during processing by the naturally greasy portions. D. W.

**Hop tannins.**—See XVIII.

#### PATENTS.

**Tanning process.** C. O. HENKE and A. C. ORTHMANN, Assrs. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,897,124, 14.2.33. Appl., 25.5.31).—Hides and skins are tanned with a mixture of a vegetable tanning agent and  $\lambda$  an equal amount of a sulphonation product of abietene-like hydrocarbons, their salts, or aldehyde condensation products as described in U.S.P. 1,853,353 (B., 1933, 157). D. W.

**Colloidal suspension and its use [in tanning].** I. C. SOMERVILLE, Assr. to ROHM & HAAS Co. (U.S.P. 1,897,773, 14.2.33. Appl., 12.7.30).—A colloidal suspension of the product obtained by condensing equimol.



proportions of naphthalenesulphonic acid and dibenzyl-naphthalene by means of  $\text{CH}_2\text{O}$  in presence of  $\text{H}_2\text{SO}_4$  of non-sulphonating concn. is claimed. D. W.

**Manufacture of gelatin capsules.** M. STEIN, Assr. to J. R. KELLER (U.S.P. 1,898,507, 21.2.33. Appl., 15.6.31. Austr., 22.5.30).—A mixture of gelatin (400 pts.), glycerin (100 pts.), petroleum (0.015–0.02 pt.), and gum benzoin (0.03–0.04 pt.) is formed into capsules and hardened with 3–5%  $\text{CH}_2\text{O}$ , EtOH, and glycerin. D. W.

**Extraction of glue from glue-containing material.** J. KOHL (U.S.P. 1,904,003, 18.4.33. Appl., 17.3.31. Ger., 8.4.30).—The liquor from each unit of a battery of boilers containing crushed bones at successive stages of extraction is pumped to an attached receptacle and the material is subjected to steam-treatment to loosen more glue; the liquor is then returned to the boiler and is forced to the next of the series by pumping hot  $\text{H}_2\text{O}$  into the one containing the most highly extracted charge. L. A. C.

**Manufacture of adhesives.** W. D. FAWTHROP (U.S.P. 1,897,469, 14.2.33. Appl., 5.2.30).—A mixture of a (de-oiled) vegetable protein flour, e.g., soya-bean flour, and < 3% of an acid-reacting medium, e.g., gallotannic acid,  $\text{ZnSO}_4$ ,  $\text{K}_2\text{Cr}_2\text{O}_7$ , is mixed with  $\text{H}_2\text{O}$  and to the mixture excess of a strongly alkaline (NaOH) solution at 80–100° is added. D. W.

**Conservation of technical preps.**—See III.

## XVI.—AGRICULTURE.

**Physical and chemical properties of the soils of the Hilton and Morton areas, Monroe County, and their relation to orchard performance.** III. J. OSKAMP and L. B. BATGER (Cornell Univ. Agric. Exp. Sta. Bull., 1933, No. 575, 34 pp.).—Soils in which the colloid fraction of the  $B_1$  horizon is > that of  $A_1$  possess satisfactory internal drainage for orchards. Where the rate of  $\text{H}_2\text{O}$ -percolation through  $A_1$  is > 2.5 times that through  $B_1$  injurious  $\text{H}_2\text{O}$ -logging is to be anticipated. High saturation with bases is associated with poor drainage of soils and low productivity, and *vice versa*. The limiting factor in orchard production in this area is slow drainage rather than fertility. A. G. P.

**Significance of surface area in phenomena of exchange adsorption.** A. N. CHARIN (Forsch. bodenk. lab. Tabakinst. Krasnodar, 1931, No. 78; Proc. Internat. Soc. Soil Sci., 1933, 8, 83–84).—The exchange capacity of soils of varied genetic type bears no relationship to the total surface area of the particles or to hygroscopicity. The adsorption capacity ( $A$ ) is largely affected by the org. fraction of the soil colloids.  $A$  of the zeolitic part of the complex is greatest in podsols, less in chernozems, and least in krasnozems, and is related to the  $\text{SiO}_2$  and sesquioxide content of the colloids. A. G. P.

**Application of the Neubauer test to the examination of tropical soils.** A. JACOB (Ernähr. Pflanze, 1933, 29, 290–295).—Under tropical conditions rye is unsuitable as a test plant. The use of rice leads to satisfactory agreement between results of Neubauer tests and that of field trials for K determinations, but is less

reliable for examination of the  $\text{PO}_4^{'''}$  status. In the case of irrigated soils Neubauer tests should be supplemented by observations of the rate of movement of  $\text{H}_2\text{O}$  in these soils. A. G. P.

**Determination of the potash requirement of arable soils.** F. NYDAHL (Kung. Landtbruks-Akad. Handl. Tidskr., 1932, 71, 845–868; Chem. Zentr., 1933, i, 2300).—Extraction with  $\text{H}_2\text{O}$ ,  $\text{N-}$  or  $0.04\text{N-NH}_4\text{Cl}$ ,  $0.5\text{N-aq. NH}_3$ ,  $\text{N-AcOH}$ ,  $0.1\text{N-CaCl}_2$ , or  $\text{Ca(HCO}_3)_2$  gave parallel results; use of  $\text{CaCl}_2$  is simplest. In 70–85% of the tests the results were in accord with those of pot and field experiments. A. A. E.

**Determination of the fertiliser requirement of soil.** I. G. SUNDELIN, O. FRANCK, and C. LARSON (Kung. Landtbruks-Akad. Handl. Tidskr., 1932, 71, 974–1004; Chem. Zentr., 1933, i, 2300).—Comparative tests on various Swedish soils show that Mitscherlich's method gives a quant. measure of the requirement. Neuburger's method is less satisfactory. A. A. E.

**Phosphate fertilisers on Montana soils.** E. BURKE, I. J. NYGARD, and W. McK. MARTIN (Montana Agric. Exp. Sta. Bull., 1933, No. 280, 52 pp.).—On irrigated soils  $\text{NH}_4$  phosphate produced better yields of potatoes which were longer, more pointed, and rougher than when treble superphosphate (I) was used. On meadows where cattle were afflicted with bone-chewing, application of (I) increased both the yield and P content of the hay. Use of P fertilisers on lucerne slightly increased the N content of the crop. A. G. P.

**Yield decrease due to addition of potassium fertilisers.** A. L. MASLOVA (Chem. Social. Agric., 1932, No. 9–10, 78–83).—Residual acidity of K salts frequently causes decreased yields; KCl is more injurious than  $\text{K}_2\text{SO}_4$ . With N, but not in a complete fertiliser,  $\text{Na}_2\text{SO}_4$  substituted for  $\text{K}_2\text{SO}_4$  is effective. CH. ABS.

**Pathological symptoms of potash starvation [in plants].** L. KRATSCHEMER (Ernähr. Pflanze, 1933, 29, 264–265).—Deficiency of K is associated with increased yellow rust in barley, drought spot in oats, and leaf roll in potato. A. G. P.

**Potash content and potash manuring of vines and soils in the Moselle, Saar, and Rumer districts.** SCHRADER (Ernähr. Pflanze, 1933, 29, 266–268).—Neubauer analyses indicate that customary manurial treatments do not maintain an optimum K supply in these soils. A. G. P.

**Influence of potash manuring on sensitiveness of apple trees to apple aphids and mildew.** II. O. JANCKE (Arb. Biol. Reichsanst. Land- u. Forstwirts., 1933, 20, 291–302).—Neither in pot-culture nor orchard trials was any relationship apparent between K manuring and the proportion of injured trees. A. G. P.

**Factors affecting the use of nitrate- and ammonium-nitrogen by apple trees.** V. A. TIEDJENS and M. A. BLAKE (New Jersey Agric. Exp. Sta. Bull., 1932, No. 547, 32 pp.).—In sand cultures of sufficiently high  $p_H$  apple trees absorbed and assimilated  $\text{NH}_4\text{-N}$  without preliminary oxidation to  $\text{NO}_3^-$ . In nutrients of low  $p_H$  the conversion of  $\text{NH}_4^+$  into  $\text{NO}_3^-$  is an essential preliminary to absorption. At  $p_H$  ranges permitting the intake



of  $\text{NH}_4^+$  and  $\text{NO}_3^-$  the former produces the more rapid growth responses and, during assimilation, the more rapid formation of org. N material (notably sol. N substances) in the plant. The hemicellulose content was consistently higher in those trees receiving  $\text{NH}_4^+$  at  $p_{\text{H}}$  ranges favourable to its assimilation. Reductase activity was low in or absent from trees growing rapidly in  $\text{NH}_4^+$  nutrients. Assimilation of both  $\text{NO}_3^-$  and  $\text{NH}_4^+$  occurred in the fine fibrous roots. The greatest vol. of growth occurred in trees in which the ratio protein-N : sol. N was high.

A. G. P.

**Effects of varying amounts of nitrogen on the growth of tulip poplar seedlings.** A. G. CHAPMAN (Ohio J. Sci., 1933, 33, 164—181).—The tissues contained little  $\text{NO}_3^-$ -N regardless of the  $\text{NH}_4\text{NO}_3$  concn. in the medium. EtOH-sol. N was much < EtOH-insol. N. Total N decreased in the order: leaf, root, bark, woody cylinder. The relation of N to dormancy and growth was studied.

CH. ABS.

**Imbibition of nutrients by plants and the return of these by plants to the soil in relation to the conditions of growth.** N. S. AVDONIN (Chem. Social. Agric., 1932, No. 9—10, 51—67).—At maturity oats return org. and inorg. substances to the soil. The quantity of ash constituents returned depends on the intake during the period of active growth. The highest return takes place from the straw when it begins to dry out and transpiration is high.  $(\text{NH}_4)_2\text{SO}_4$  exerts a solubilising effect on raw phosphate in the early stages.

CH. ABS.

**Correcting the unproductiveness of acid and alkaline muck soils for the growing of vegetable crops.** G. M. TAIT and J. E. KNOTT (Cornell Univ. Agric. Exp. Sta. Bull., 1933, No. 572, 19 pp.).—Artificial adjustment of the reaction of these soils is examined. The optimum  $p_{\text{H}}$  range for various crops grown on initially acid soil after liming differed from that on initially alkaline soil after treatment with S. Crop yields were not appreciably affected by changes of soil reaction in the range  $p_{\text{H}}$  5.0—7.0.

A. G. P.

**Amount of plant food removed from soil by leguminous crops, as based on their nutrient contents.** W. KLEBERGER and H. RUDOLF (Ernähr. Pflanze, 1933, 29, 241—244).—Analyses of various crops are recorded. Except in the case of horse beans and lupins, the ratio of the various mineral nutrients removed from soil is approx. the same for all leguminous plants.

A. G. P.

**Vine growth and soil reaction.** K. MÜLLER and H. SLEUMER (Weinbau u. Kellerwirts., 1933, 12, 11—14; Chem. Zentr., 1933, i, 2299).—With a soil mixture and with sand + nutrient solution optimum growth occurred at  $p_{\text{H}}$  6.1.

A. A. E.

**Relation of the tobacco plant to essential plant-food elements.** A. V. OTRUGANIEV (Chem. Social. Agric., 1932, No. 9—10, 68—77).—An increase in N fertilisers increases the N and nicotine contents of the tobacco and decreases the carbohydrate content in a loam chernozem.  $\text{P}_2\text{O}_5$  increases the yield, but not the  $\text{P}_2\text{O}_5$  content, of the tobacco. Lack of K decreases the yield < lack of N. With an increase in K the Ca content of the tobacco increases.  $\text{K}_2\text{SO}_4$  and  $\text{K}_2\text{CO}_3$  are

superior to KCl. Ca increases the yield on acid soils but does not affect the quality.

CH. ABS.

**Fundamental theory and the law of diminishing returns in agriculture.** A. H. HOLLMANN (Landw. Jahrb., 1933, 77, 689—708).—A review and discussion.

A. G. P.

**Ensilage with the "Aurich system" steel silos.** A. GOLF and K. GNEIST (Bied. Zentr. [Tierernährung], 1933, B, 5, 372—397).—In young clover silage prepared by cold fermentation in steel silos acid production was small although  $p_{\text{H}}$  4.0 was reached. With the exception of 10% N-free extractives, nutrient losses were small. When the Fingerling (HCl) process was used, acid formation in the silage was extremely small (no  $\text{PrCO}_2\text{H}$ ). There was a transition of protein to amides, but a proportion of undigestible protein was changed into a digestible form.

A. G. P.

**Nematode infestation symptoms on barley as a means of determining the efficiency of chemicals as lethal agents against *Tylenchus dipsaci*.** Kuhn. W. NEWTON, R. J. HASTINGS, and J. E. BOSHER (Canad. J. Res., 1933, 9, 37—42).—Among numerous substances examined, only PhOH,  $\text{AgNO}_3$ ,  $\text{NaHSO}_3$ , and  $\text{KHSO}_3$  were sufficiently toxic at small concns. for effective use against the nematode. The survival of nematodes was examined by growth of barley in sterile soil inoculated with treated material.

A. G. P.

**Relation of environmental factors to the occurrence and severity of blast disease in rice plants.** T. HEMMI (Phytopath. Z., 1933, 6, 305—324).—Diseased plants were more numerous in dry than in humid soils. Humidity favours the development of mechanical tissue and the accumulation of silicates in the epidermal cells. Susceptibility to disease is associated with the absorption of silicates from soil.

A. G. P.

**Possible changes in the wax-like coating of apples caused by certain spray and other treatments.** K. S. MARKLEY and C. E. SANDO (Plant Physiol., 1933, 8, 475—478).—Examination of the ursolic acid and oily fraction of the wax covering of unsprayed and oil-sprayed apples indicates that oil stimulates the natural production of the waxy substance. The increased difficulty in removal of spray residues after applications of oil is due to this fact rather than to the supposed retention of sprayed oil on the cuticle.

A. G. P.

**Copper seed treatments for control of damping-off of spinach.** P. P. PIRONE, A. G. NEWHALL, W. W. STUART, J. G. HORSFALL, and A. L. HARRISON (Cornell Univ. Agric. Exp. Sta. Bull., 1933, No. 566, 25 pp.).—Best results were obtained by soaking seed in 1% aq.  $\text{CuSO}_4$  for 1 hr., or shaking with  $\text{Cu}_2\text{O}$  till seed are well coated.

A. G. P.

**Weed control in asparagus planting.** R. M. SMOCK (Ohio Agric. Exp. Sta. Bimonth. Bull., 1933, No. 164, 124—125).—Applications of  $\text{CaCN}_2$  at the rate of 500 lb. per acre, when weeds were 1—2 in. high, effectually controlled annual weeds throughout the cutting season. Granular and pulverised forms were equally effective.

A. G. P.

**Control of ragwort on grassland. Spraying and dry-dusting methods of using sodium chlorate.**



J. W. DEEM (New Zealand J. Agric., 1933, 47, 99—104).—Spraying proved slightly the more effective method. For dusting methods  $\text{CaCO}_3$  was the most satisfactory carrier. A. G. P.

**Sterilisation of narcissus bulbs by immersion in silver nitrate-potassium cyanide solution *in vacuo*.** W. NEWTON, R. J. HASTINGS, and J. E. BOSHER (Canad. J. Res., 1933, 9, 31—36).—An evacuation process is described for forcing into the bulb tissue a solution containing 0.05%  $\text{AgNO}_3$  and 0.15% KCN. Satisfactory control (96%) is obtained without appreciable injury to the bulbs. A. G. P.

**Extraction of humic acids.**—See II. HCN [as fumigant]. Polyhalite and fertilisers.—See VII.

See also A., Oct., 1018, **Decomp. of  $\text{H}_2\text{O}_2$  by soils.** 1022, **Determination of  $\text{H}_2\text{O}_2$ .** 1024, **Colorimetric determination of Na.** 1031—2, **Soils (various).**

#### PATENTS.

**Application of aqua-ammonia to fertiliser mixtures.** E. W. HARVEY, Assr. to BARRETT Co. (U.S.P. 1,894,767, 17.1.33. Appl., 1.12.28).— $\text{CaH}_4(\text{PO}_4)_2$  is spread in a thin layer and sprayed with aq.  $\text{NH}_3$  while being conveyed. B. M. V.

**Manufacture of dust disinfectants.** M. ENGELMANN, Assr. to E. I. DU PONT DE NEMOURS & Co. (U.S.P. 1,890,774, 13.12.32. Appl., 20.12.27).— $\text{HgCl}_2$  or  $\text{Hg}(\text{OAc})_2$  is intimately ground with kaolin, kieselguhr, or  $\text{Ca}(\text{OH})_2$  and an aromatic compound, e.g.,  $\text{C}_6\text{H}_4\text{Cl}\cdot\text{OH}$ ,  $\text{NH}_2\text{Ph}$ , or o-cresol. A. R. P.

**Spreader for spray compositions [insecticides etc.].** J. F. LITTOOY, Assr. to HERCULES GLUE Co. (U.S.P. 1,898,673, 21.2.33. Appl., 23.9.29).—The material comprises, e.g., casein 4—8, a deflocculator (glue) 1—2, and  $\text{Ca}(\text{OH})_2$  90—95%. L. A. C.

**Agent for exterminating animal or vegetable pests.** K. MARX and K. BRODERSEN, Assrs. to WINTHROP CHEM. Co., INC. (U.S.P. 1,898,554, 21.2.33. Appl., 21.12.28. Ger., 24.2.27).—The use is claimed of mixtures of  $\text{H}_2\text{O}$ -sol. sulphonates of hydrocarbon fractions from lignite tar oil with aromatic hydrocarbons, phenols, or naphthols. L. A. C.

**Bacterial product [for treating seeds].** F. M. CORNELL, Assr. to STIMULANT LABORATORIES, INC. (U.S.P. 1,891,750, 20.12.32. Appl., 16.6.28).—Claim is made for a powder containing *Bact. radicola*, kieselguhr (85), C black (15), starch (8 pts.),  $\text{CaCl}_2$  (1), K alum (0.5), and  $\text{NaHCO}_3$  (1 pt.), together with 25% of moisture.

**Fumigant.**—See XXIII.

#### XVII.—SUGARS; STARCHES; GUMS.

**Processing of sugar beet by the Komers and Cuker method.** K. SMOLEŃSKI (Prace Centr. Lab. Cukrownicz., 1928—1931, 23—25; Chem. Zentr., 1933, i, 2474).—The process is criticised. A. A. E.

**Purification of [beet] juice by Teatini's process.** K. SMOLEŃSKI and M. WERKENTHIN (Prace Centr. Lab. Cukrownicz., 1928—1931, 29—52, 53—97; Chem.

Zentr., 1933, i, 2475).—Experience and advantages of the process are described. The  $\text{CaO}$  requirement is about 1% of the wt. of beet. The juices and products contain more Ca salts than normally. A. A. E.

**Microflora of fermenting beet juices from a Soviet beet-sugar factory.** A. I. ROKITZKAYA and A. P. VASILIEV (Nauk. Zapiski Tzuk. Prom., 1932, 26, 59—86).—Most of the bacteria (aërobes, facultative anaërobes, and thermophilic) decomposed sucrose with the formation of acid alone or with gas. Cultures were very resistant to temp. and disinfectants. CH. ABS.

**Molasses colloids.** K. SMOLEŃSKI and A. BRODOWSKI [with I. WAISÓWNA] (Prace Centr. Lab. Cukrownicz., 1928—1931, 413—431; Chem. Zentr., 1933, i, 2475—2476).—Molasses contains considerable quantities of surface-active colloids; surface tension is max. at  $p_H$  7.0 approx. The viscosity is not  $>$  that of sucrose solutions of the same  $d$ . The colloids (0.90%) were fractionated by exhaustive dialysis. The non-reversible colloids are acid or amphoteric, dark brown, and contain 7.5—8.7% N. The reversible colloids are neutral, light brown, and have a low N content; they contain much araban and probably also dextran. The viscosity of the colloid solutions is much  $>$  that of sucrose solutions. A. A. E.

**Peculiar precipitate in a turbo-compressor for saturation gas.** K. SMOLEŃSKI and W. REICHER (Prace Centr. Lab. Cukrownicz., 1928—1931, 160—175; Chem. Zentr., 1933, i, 2474).—The composition of the ppt., which arose from the presence of insufficient air and excess of coke in the limekiln, is recorded. A. A. E.

**Characteristics and evaluation of Polish white sugar.** K. SMOLEŃSKI (Prace Centr. Lab. Cukrownicz., 1928—1931, 249—265; Chem. Zentr., 1933, i, 2476). A. A. E.

**Yeast-growth stimulants in white sugars.** H. H. HALL, L. H. JAMES, and L. S. STUART (Ind. Eng. Chem., 1933, 25, 1052—1054).—Substances in sucrose which stimulate the growth of yeast contain N, are org., and are sol. in 80% EtOH. E. C. S.

**Rapid commercial analysis of crude potash from beet vinasses.** K. DÖMÖTÖR (Z. Spiritusind., 1933, 56, 195).—In the crude residue ("Schlempekohle"), total  $\text{CO}_3$  is determined directly as  $\text{CO}_2$ , total K as  $\text{KClO}_4$ , and  $\text{K} + \text{Na}$  as chloride, or Na may be determined directly. E. S. H.

**EtOH from wood. Fermentation of starch-rich materials.**—See XVIII.

See also A., Oct., 1012, **Swelling of potato starch.** 1022, **Sb electrode for  $p_H$  determinations.** 1037, **Removal of sugars from dil. solutions.** **Determination of glucose.** **Sugar analysis.** 1038, **Cinnamyl esters of starch.**

#### XVIII.—FERMENTATION INDUSTRIES.

**Influence of oxygen on fermentation.** K. BENGTSSON (Svensk. Bryggarefören. Månadsbl., 1932, 47, 331—337; Chem. Zentr., 1933, i, 2326).—Small additions of  $\text{O}_2$  enhance the fermenting power of yeast, but large additions adversely affect it. A. A. E.



**Influence of various alkaloids on the growth and reproduction of saccharomycetes.** G. MEZZADROLI and A. AMATI (*Ind. saccharif. ital.*, 1932, **25**, 392—394; *Chem. Zentr.*, 1933, i, 2422).—Caffeine (I), quinine sulphate (II), and strychnine nitrate (III) (in order of diminishing activity; 0.025—0.1%), when added to molasses wort, increase the yield of yeast. The fermentative power of yeast is increased by (II), diminished by (III), and may be extinguished by (I).

A. A. E.

**Decomposition of pentosans in the fermentation of beer wort with yeast.** A. HEIDUSCHKA and J. SEGL (*Z. ges. Brauwes.*, 1933, **56**, 1—4, 5—8, 9—12; *Chem. Zentr.*, 1933, i, 2326).—Only a fraction of malt pentosans passes into the wort. Whilst pure arabinose alone or in presence of maltose in sterile solution is not attacked by yeast, pentosans in wort undergo considerable decomp., particularly when the acidity is high. The decomp. reaches about 35% under sterile and 50% under non-sterile conditions.

A. A. E.

**Preservative principles of hops. XV. Gravimetric determination of the antiseptic constituents of hops.** T. K. WALKER and J. J. H. HASTINGS (*J. Inst. Brew.*, 1933, **39**, 509—512; cf. B., 1933, 203).—The simplifying modification of the method of Ford and Tait (B., 1929, 533) gives too high a val. for  $\alpha$ -resin, due to the presence of a non-antiseptic substance. This is removed in the revised method by extracting the soft resins with petroleum spirit prior to pptn. by Pb(OAc)<sub>2</sub>. The results now agree with the modified Ford-Tait method (B., 1932, 858).

R. H. H.

**Distribution of the tannin and bitter principles in hops.** H. WILDNER (*Woch. Brau.*, 1933, **50**, 289—294).—The average tannin content of the hops examined was 6%, of which 89% was contained in the bracts (I), 7.4% in the hop meal (II), 1.9% in the cone axes (III), and 1.7% in the cone stems (IV). Of the total resin, amounting to 18—21% of the hop dry wt., (I) contained 21.02%, (II) 77.14%, (III) 1.13%, and (IV) 0.71%.

R. H. H.

**Modern chips in practice.** G. JACOB (*Woch. Brau.*, 1933, **50**, 285).—The vals. for attenuation (*A*) quoted by Nius (B., 1933, 888) are 9% too high. The *A* vals. in table 2 for the 8th and 9th days of fermentation should be interchanged, since a decrease is impossible.

R. H. H.

**Modern chips in practice.** H. FINK (*Woch. Brau.*, 1933, **50**, 285).—A crit. review of the experiments of Nius (B., 1933, 888).

R. H. H.

**Pitched plate-chips.** ECKERT (*Woch. Brau.*, 1933, **50**, 277—278).—Contrary to the results of Hell (B., 1933, 681), pitched Al foil is superior to Mammut bio-chips since the latter accelerate the autolysis of the yeast and may lead to deterioration in the flavour of the beer. Beer is not contaminated by contact with Al.

R. H. H.

**Institute of Brewing. Standard methods of analysis.** ANON. (*J. Inst. Brew.*, 1933, **39**, 517—525).—The 1933 revised methods adopted by the Institute in the analysis of malts, flaked maize and rice, grits, and raw grains are detailed. The prep. of reagents is also described.

R. H. H.

**Protein changes during mashing.** P. KOLBACH and R. BUSE (*Woch. Brau.*, 1933, **50**, 265—270, 273—277, 281—285).—The proteins of worts are differentiated by adsorption on active C (I), on tannin (II), and by pptn. by MgSO<sub>4</sub> (III). (I) adsorbs mainly colloidal protein of high mol. wt. (IV), (II) adsorbs coagulable protein, some albumose, and peptones (V), and (III) ppts. dissolved protein of high mol. wt. (VI). At the same mashing temp. (IV) increases with the duration of mashing. With equal periods of mashing and varying temp. (IV) and (V) show a max. at 60—65°, whilst (VI) increases up to 70°, due in part to synthesis. The increase of colloidal N compounds at higher mashing temp. is due to the greater heat-susceptibility of the peptidase than of the proteinase, which produces (V) from insol. protein. As % permanently sol. N, the N of fractions (II) and (III) decreases at all temp. with increase of *pH*, whilst with U(OAc)<sub>4</sub> and (I) adsorbates the influence of reaction is less clear. A shift from the optimum *pH* of the proteinase increases the abs. amount of (V), which is so rapidly degraded that the N of (II) and (III) as % permanently sol. N decreases, and the intermediate products of protein degradation increase. (IV) increases with the concn. of the mash. The protective action of conc. mashes diminishes the heat-inactivation of the peptidase, and leads to a relative increase of low-mol. degradation products. R. H. H.

**Proposed official international method for the analysis of wines.** G. FILAUDEAU (*Ann. Falsif.*, 1933, **26**, 420—423).—A report to the French Committee in which a plea is made for an international method of expression of results, and methods are outlined for rapid sorting purposes and for complete analysis in the following cases: *d*, EtOH, solids, total acidity, H<sub>2</sub>SO<sub>4</sub>, ash, microscopical examination (the same methods in both cases); reducing sugars, SO<sub>3</sub><sup>2-</sup>, K<sub>2</sub>O, tartaric acid, preservatives (qual. only in the former case). J. G.

**Determination of tartaric acid in musts and wines.** L. SEMICHON and M. FLANZY (*Ann. Falsif.*, 1933, **26**, 403—406).—The ppt. produced in a mixture of 5 c.c. each of wine and 2% NH<sub>4</sub> l-tartrate and 50 c.c. of saturated aq. CaSO<sub>4</sub> is boiled, filtered cold and washed the next day, and a suspension in dil. H<sub>2</sub>SO<sub>4</sub> titrated with KMnO<sub>4</sub> at the b.p. Comparison with existing methods (cf. B., 1932, 814) for synthetic mixtures showed that this gives the most reliable results. J. G.

**Radioactivity of musts and wines.** E. CANALS and A. MÉDAILLE (*J. Pharm. Chim.*, 1933, [viii], **18**, 155—156).

—The radioactivity (*R*) of a new wine corresponds to the total of those of the must and the marc. There is no relationship between *R* and the course of the fermentation. 3 wines, after 8—20 days' fermentation, had activities of 0.104—0.200 millimicrocurie (cf. B., 1932, 859).

E. H. S.

**Determination of sulphates in wines by the benzidine method.** E. LOBSTEIN and M. ANGEL (*Ann. Chim. Analyt.*, 1933, [ii], **15**, 389—397).—A mean error of <1.5% results if Raschig's method is modified (to avoid interference by org. acids, P<sub>2</sub>O<sub>5</sub>, CO<sub>2</sub>, EtOH, etc.) by pptg. 50 c.c. of neutral wine containing 5 c.c. of 0.1N-HCl with 30 c.c. of 40% benzidine hydrochloride, the ppt. being washed with Et<sub>2</sub>O + EtOH, suspended



in  $H_2O$ , and titrated (phenolphthalein) with 0.1N-NaOH; if the wine solids are first incinerated in a pyrex vessel the error is  $< 1\%$ . For the control method, pptn. of a solution of the ash is preferred to pptn. of the wine direct, and gives results 0.9–14% higher. J. G.

**Colorimetric determination of iron in red wines.** J. DUBAQUIÉ (Ann. Falsif., 1933, 26, 418–420).—Polemical against and a claim for priority over Ribéreau-Gayon (cf. B., 1933, 601, 729). The determination of total Fe by addition to the wine of HCl, KCNS, and  $H_2O_2$  and matching an extract in  $Et_2O$  of the resulting colour against similarly prepared standards is only approx., even if allowance is made for the EtOH content by appropriate additions to the standards. For  $Fe^{+++}$ , peroxide-free  $Et_2O$  should be used. J. G.

**Distinction of wine vinegar from other types of vinegar.** A. PATZAUER (Chem.-Ztg., 1933, 57, 735).—10 c.c. of filtered sample are shaken first with 1 c.c. of a 20% KOAc solution (I), and then with 0.5 c.c. of a solution prepared by pptg. 0.3 c.c. of (I) with a mixture of 0.2 g. of l-tartaric acid and 0.4 g. of NaOAc in 20 c.c. of  $H_2O$  and diluting the filtrate to 100 c.c. Wine vinegars give a deposit only after a few min. to 1 hr., microscopical examination of which then provides an indication of the degree of fermentation and the nature of the starting product. J. G.

**Preparation of [ethyl] alcohol from wood.** L. I. ANTZUS (J. Appl. Chem. Russ., 1933, 6, 705–709).—34% yields of reducing sugar (I) are obtained by hydrolysing pine sawdust (II) during 48 hr. with 37% HCl in presence of  $ZnCl_2$  [2 g. per g. of (II)]. The yield is not increased by using more conc. HCl. (I) is readily fermentable, 340 c.c. of EtOH being obtained per kg. of (II). Small quantities of AcOH are found amongst the products of hydrolysis of (II). R. T.

**Butyric acid and butyl alcohol fermentation of hemicellulose- and starch-rich materials.** S. A. WAKSMAN and D. KIRSH (Ind. Eng. Chem., 1933, 25, 1036–1041).—Two strains of anaerobic bacteria of the *Clostridium butyricum* group, grown on hemicellulose-containing media, produced large amounts of  $PrCO_2H$  (I) and small amounts of non-volatile acid, including lactic acid. One of them produced a typical BuOH (II) fermentation. Wheat middlings were fermented readily, maize meal was not. Addition of maize steep improved the fermentation of both. Addition of  $CaCO_3$  increased the yield of (I), diminished that of (II). In other substrates the extent of fermentation varied with the amount of available carbohydrate. Although the organisms were able to utilise pentosans in the form of corn-cobs, mannans in the form of salep root, and galactan in the form of Irish moss, very little growth occurred on the purified hemicelluloses. Casein was better than were  $NH_4$  salts as the source of N. E. C. S.

**Rectification.**—See I. **Dehydrating EtOH.**—See III. **Vineyard soils.**—See XVI.

See also A., Oct., 1037, **Removal of sugars from dil. solution.** 1080–2, **Enzymes (various).** 1082, **Action of coal tar and ultra-violet rays on yeast development.** Prep. of  $H_2C_2O_4$  from  $HCO_2H$ . 1082, **Bacterial production of amyl alcohol.** 1083,

**Fermentation of cellulose, and of polyhydric alcohols by bacteria.**

#### PATENTS.

**Malting of grain.** R. E. KUSSEROW (U.S.P. 1,899,160, 28.2.33. Appl., 12.5.31).—The grain is bedded on a metal screen supported in a malting tank. The steeping and germination (I) are carried out in the same tank and the temp. maintained correct in (I) by alternately forcing moistened air up, or dry air down, through the bed of grain. R. H. H.

**Manufacture of alcohol-weak beverages.** A. LEWINSON (U.S.P. 1,899,512, 28.2.33. Appl., 8.8.30. Ger., 9.10.29).—In the presence of cranberry extract, fermentation of sugar by yeast proceeds only to a small extent and the beverage can be stored without any appreciable secondary fermentation taking place. Flavouring, e.g., vanilla, may be added if desired. R. H. H.

**Manufacture of distillable spirits by fermentation.** S. D. WELLS (U.S.P. 1,904,589, 18.4.33. Appl., 17.6.31).—The liquor obtained by cooking fibrous material with mild cooking agents, e.g.,  $Na_2SO_3$ ,  $Na_2CO_3$ , or CaO, is rich in carbohydrate and may be readily acidified and fermented by suitable bacteria. The flow-sheet of a process in which *Clostridium acetobutylicum* is the fermenting organism and  $COMe_2$ , BuOH, and EtOH are the products is given. R. H. H.

**Unfermented milk products.**—See XIX.

#### XIX.—FOODS.

**Determination of the bitter substance in "bitter-weed" milk.** N. D. WEATHERS (J. Dairy Sci., 1933, 16, 401–403).—Milk contaminated with the bitter principle of *Helenium tenuifolium* gives a characteristic orange-red colour when treated successively with solutions of picric acid (1.2%) and NaOH (10%). For quant. work an EtOH- $Et_2O$  extract of centrifuged milk is prepared for analysis and standard colours are obtained from purified bitter crystals or creatinine or picramic acid standards. A. G. P.

**[Electrical] sterilisation of liquids.** E. A. LOUDER, Assr. to PET MILK Co. (U.S.P. 1,900,509, 7.3.33. Appl., 30.10.30).—A stream of milk is projected across the gap between two electrodes, e.g., a nozzle and a plate, maintained at a p.d. of about 6000 volts, so as to strike against the second electrode. J. S. G. T.

**Fungi found in butter.** G. R. BISBY, M. C. JAMIESON, and M. TIMONIN (Canad. J. Res., 1933, 9, 97–107).—The kinds of fungi found in samples of butter produced in Manitoba during 1932 are enumerated. In most cases these are also present in the soil or in decaying vegetable matter. Precautions necessary for the manufacture of mould-free butter are discussed. Moulds grow less readily in salted butter. A. A. L.

**Whipping cream.** H. L. TEMPLETON and H. H. SOMMER (J. Dairy Sci., 1933, 16, 329–345).—Optimum whipping properties of cream are associated with a fat content of 30–34%. Poor whipping capacity in early spring cream is improved by the addition of 0.1–0.4% of Na citrate (I). (I) additions also decrease the whipping time of creams containing  $< 34\%$  of fat. A. G. P.



**Surface tension of homogenised cream.** B. H. WEBB (J. Dairy Sci., 1933, 16, 369—373).—The surface tension of homogenised cream increased with the pressure of homogenisation and with the fat content. A. G. P.

**Early abnormal fermentation of cheese.** E. HAGLUND, E. SANDBERG, and C. BARTHEL (Kung. Landtbruks-Akad. Handl. Tidskr., 1932, 71, 801—822; Chem. Zentr., 1933, i, 2331).—An apparatus for the determination of  $\text{CO}_2$  and  $\text{H}_2$  (evolved during the first four days) is described. By the simultaneous addition of starters and  $\text{KNO}_3$  to milk, or by pasteurisation at  $63^\circ$  for 10 min., gas production by *B. aerogenes* may be completely suppressed. The salts present in cheese favour gas production. A. A. E.

**Variable composition of casein and its rôle in the manufacture of cheese.** E. CHERBULIEZ (Arch. Sci. phys. nat., 1933, [v], 15, Suppl., 156).—Of two cow's milks, that containing the higher proportion of the  $\alpha_2$  constituent (cf. A., 1933, 843) of casein coagulated the more readily. R. S. C.

**Beating properties of egg-white.** W. C. HENRY and A. D. BARBOUR (Ind. Eng. Chem., 1933, 25, 1054—1058).—Methods of determining foam vol. (I) and foam stability (II) of beaten egg-white are described. (I) of thin white of egg is at first  $>$  that of thick white, but diminishes when beating is continued. Storage for 1 month in the frozen state has no effect on (I), but (II) is increased. Addition of  $\text{H}_2\text{O}$  up to 80% causes an increase in (I) calc. on the egg-white content, but lessens (II). Addition of cottonseed oil up to 1% lessens (I) and (II). (I) increases with increase in  $p_{\text{H}}$ , particularly above  $p_{\text{H}}$  10. (II) is at a max. between  $p_{\text{H}}$  5.47 and 8, and at a min. between  $p_{\text{H}}$  8 and 10. E. C. S.

**Under what conditions are calcium and phosphorus supplements needed in the feeding of farm animals?** G. BOHSTEDT (Proc. Amer. Soc. Animal Produc., 1932, 298—302).—Ca and P deficiencies are best overcome by proper fertilisation of the forage crops. CH. ABS.

**Feeding value of *Artemisia sieversiana* (wormwood).** V. I. KOPUIRIN (Trans. Omsk Inst. Dairying, 1931, 1, No. 1, 49—57).—The plant contains protein 15.5 and fat 5.12%, the digestive coeffs. being 62.2 and 71.4%, respectively. The santonin content is low. CH. ABS.

**Composition and digestibility of silage from wormwood (*Artemisia sieversiana*) in relation to its stage of growth and methods of silage-making.** P. A. KORMSHIKOV (Trans. Omsk Inst. Dairying, 1932, 2, No. 1, 19—49).—Silage from wormwood in the early stage contained (dry wt.) protein 15—19.3 and fat 3.46—6.91%. The protein content was higher and the fat content lower in silage made from young plants by the cold method than in that made by the hot method. Mature plants gave a lower albumin content but a higher fat and protein content by the cold method. The digestibility of silage from young plants was 55.17% (cold), 49.54% (hot), and from plants cut in bloom 48.78 and 47.57%, respectively. CH. ABS.

**Influence of silage from wormwood (*Artemisia sieversiana*) on the physico-chemical properties**

**of butter.** V. I. KOPUIRIN (Trans. Omsk Inst. Dairying, 1932, 2, No. 1, 83—95).—Milk-taste and -lactose were unaffected. Silage from the bloom stage did not affect butter const.; that from the fruit stage decreased the Reichert-Meissl val. and increased  $n$ . CH. ABS.

**Digestibility of clover and lucerne.** P. A. KORMSHIKOV (Trans. Omsk Inst. Dairying, 1931, 1, No. 1, 33—47).—The digestive coeffs. of the fat were, respectively, 58.17, 49.48%; the protein coeffs. were 72.48, 64.63%, N-free extract coeffs. 64.80, 64.85%, and cellulose coeffs. 31.68, 48.15%. CH. ABS.

**Seasonal variation in composition and digestibility of certain species of range bunch grasses.** R. MCCALL (Proc. Amer. Soc. Animal Produc., 1932, 95—100).—Changes in composition with maturation of *Festuca idahoensis* are recorded. The grass is low in digestible ash and crude protein, but the digestibility of the crude fibre is high, and that of the N-free extract and fat is fairly high. CH. ABS.

**Composition of early pasture legumes and grasses.** R. H. LUSH (Proc. Amer. Soc. Animal Produc., 1932, 91—94).—*Trifolium repens*, *Lolium multiflorum*, *Poa annua*, oats, and *T. dubium* grown in Louisiana had high protein and  $\text{H}_2\text{O}$  and low fibre contents. CH. ABS.

**Factors influencing the vitamin-B and -G content of hays.** C. H. HUNT, P. R. RECORD, W. WILDER, and R. M. BETHKE (Ohio Agric. Exp. Sta. Bimonth. Bull., 1933, No. 163, 104—106).—Losses of protein (I) and vitamin-G (II) occur during the curing of hay. Normally a high (I) content and good green colour are associated with high (II) activity. Rain removes proportionally more (II) and colour than (I). A. G. P.

**Green forage and methods of conservation.** II, III. **Marrowstem kale.** Silage of marrowstem kale and swede leaves and tops. H. EDIN, N. BERGLUND, and Y. ANDERSON (Repts. Nos. 431 and 433, Centr. Agric. Res. Stat., Stockholm, 1933).—II. The protein content of clover aftermath (A) is so high (14—17 kg. of digestible true protein per 100 kg. of org. matter) that it cannot be efficiently utilised even by high-milking cows. Marrowstem kale (B) (9—13 kg. of digestible true protein per 100 fodder units) should be used during Sept. and Oct. and the A reserved for winter use as silage. The effect on consistency of butter would in both cases be advantageous. A thoroughly compacted in airtight silos does not require addition of HCl or sugar to give optimum acidity, but under other conditions addition of HCl or sugar prevents  $\text{Pr}\cdot\text{CO}_2\text{H}$  fermentation. Crops dried at  $600^\circ$  with very short exposure show satisfactory vitamin-A content.

III. The properties of B are not affected by harvesting at any time between mid-Sept. and Dec., but thinning out has a marked beneficial effect on the val. of the crop. 50—60 kg. per head per day fed to dairy cows ensure optimum consistency of butter, and the flavour of milk is unaffected by 55 kg.

Data for composition, digestibility, and feeding val. of silage from B and swede tops and leaves are given. Ensiling does not appear to reduce digestibility.

NUTR. ABS. (m)



**Feeding trials with marrowstem kale.** H. BÜNGER [with A. WERNER, P. GLET, J. SCHULTZ, and J. KESELING] (Bied. Zentr. [Tierernährung], 1933, **B**, 5, 450—468).—Digestibility data are recorded. Stems were poorer than leaves in crude protein and ash and richer in crude fibre and N-free extractives. When fed in moderate amounts (40 kg. daily) towards the end of the grazing season, kale increase the milk yield with a slight reduction in % milk fat. Larger amounts adversely affected the flavour of the milk. A. G. P.

**Vitamin-C content of strawberries and strawberry ice cream.** C. R. FELLERS and M. J. MACK (Ind. Eng. Chem., 1933, **25**, 1051—1052).—Fresh strawberries are rich in vitamin-C. There is no loss on freezing, during storage in the frozen state, either with or without sugar, or as a result of incorporation in ice cream. E. C. S.

**Effect of manufacturing and preserving processes on the vitamins of cranberries.** P. D. ISHAM and C. R. FELLERS (Mass. Agric. Exp. Sta. Bull., 1933, No. 296, 19 pp.).—The vitamin-C content of cranberries, which was high in fresh fruit, was markedly affected by certain preserving processes. Vitamins-B, -D, and -G were present in the fruit only in insignificant amounts. A. G. P.

**Food analysis and wet oxidation with perchloric acid.** L. BORNAND and (MLLE.) M. R. DUMONT (Ann. Falsif., 1933, **26**, 406—408).—For > 10 g. of sample, or for fatty substances, preliminary oxidation is achieved by  $\text{HNO}_3 + \text{H}_2\text{SO}_4$  (5:1) and completed by addition dropwise of commercial 6% aq.  $\text{HClO}_4$  (*d* 1.61), which is quite stable. In other cases  $\text{HNO}_3 + \text{HClO}_4$  (3:1) is used. J. G.

**Herring-meal fat. Vitamin-D of cod-liver oil. Halibut-liver oil.**—See XII. Fish-preserving oils. —See XII.

See also A., Oct., 1968, Action of NaCNS on milk.

#### PATENTS.

**Making a therapeutic milk product.** M. C. MATT, Assr. to ABBOTTS DAIRIES, INC. (U.S.P. 1,899,817, 28.2.33. Appl., 23.5.31).—Milk containing viable *B. acidophilus* in therapeutically effective amounts and having an acidity of 0.6—0.7% expressed as lactic acid is mixed with cultured milk containing *S. lactis* and of similar acidity. E. H. S.

**Unfermented (A) acidophilus milk, (B) milk product.** R. P. MYERS, Assr. to RESEARCH LABS. OF NAT. DAIRY PRODUCTS CORP., INC. (U.S.P. 1,902,959 and 1,903,014, 28.3.33. Appl., [A] 16.4.30, [B] 21.10.31).—*Lactobacillus acidophilus* is grown in rennet whey, or a solution of rennet-whey powder, to which has been added peptone, tomato juice, malt syrup, or wheat-germ extract, and carbohydrate to 10%; the cells are centrifuged or filtered off, washed free from acid, and the viable concentrate is sold either in tablet form or as a suspension in homogenised milk, when the temp. is kept below 10°, in the correct therapeutically active doses. R. H. H.

**Paper [for foodstuffs].**—See V. Inhibiting rancidity of fats etc.—See XII. EtOH-weak beverages.—See XVIII.

#### XX.—MEDICINAL SUBSTANCES; ESSENTIAL OILS.

**Ageing of cresol soap solution and aluminium acetate.** O. SCHMATOLLA (Pharm. Ztg., 1933, **78**, 870—871).—Freshly prepared cresol soap solution made with D.A.B. IV K soap (I) does not immediately form a clear solution in  $\text{C}_6\text{H}_6$ , whereas D.A.B. VI K soap (II) does. This is probably due to a structure alteration of (I) with change in  $\text{H}_2\text{O}$  distribution between the soap and the cresol. If K soap with 60% oil content be used and then the necessary  $\text{H}_2\text{O}$  added, the prep. is sol. immediately.  $\text{Al}(\text{OAc})_3$  solutions prepared by dilution [to (II) concn.] of conc. solutions (21—22%) deposit almost all their subacetate (III) content in a few months, whereas those prepared at the correct concn. (over  $\text{CaSO}_4$ ) show only the normal cloudiness. This again is due to differences in  $\text{H}_2\text{O}$  distribution; the conc. solution contains a different form of (III) (a polymeride which retains its structure on dilution), which is less sol. It is not possible to prepare stable solutions of concn. > 7.5—8.0% of (III) and *d* 1.041—1.043. E. H. S.

**Presence and conservation of biological properties in organic powders.** A. CHOAY (J. Pharm. Chim., 1933, [viii], 18, 137—144).—The technique of the French Codex for the prep. of biological powders adapted to hormone preps. gives products having activities equiv. to the original material and remaining stable when protected from air and moisture. This is exemplified by experiments described with thyroid, suprarenal, and ovarian glands, posterior-lobe extracts, and pancreatic amylase. Hepatic catalase and amylase and insulin showed losses of activity, but after 6—8 years these substances were still appreciably active. E. H. S.

**Use of diphenylamine in determining small quantities of trinitrin [glyceryl trinitrate].** C. H. SYKES (Pharm. J., 1933, **131**, 267—268).—A crushed tablet [0.01 grain of trinitrin (I)] is treated with 2.5 c.c. of glacial AcOH with occasional shaking for 2 hr. and the mixture filtered. To 1 c.c. of the filtrate in a 10-c.c. graduated tube is added 0.25 c.c. of the 0.5%  $\text{NHPh}_2\text{-H}_2\text{SO}_4$  reagent. After shaking the tube immersed in cold  $\text{H}_2\text{O}$ , AcOH is added to the mark and the colour compared with that of 2.5 c.c. of standard solutions (0.0346, 0.0231, and 0.0173 g. of  $\text{KNO}_3$  in 100 c.c. of AcOH) treated similarly. Tablets made and stored with reasonable care retain 75—100% of their (I) content after storage for 2 months. E. H. S.

**Vitamin-D of cod-liver oil.**—See XII.

See also A., Oct., 1944, Local anæsthetics. 1045, Br-derivatives of thymoquinol. 1046, Urethanes of  $\alpha$ -m-hydroxyphenylalkyldimethylamines. Prep. of p-hydroxyphenylaminoacetic acid, and of Bi compounds of pyrocatechol, pyrogallol, and gallic acid. 1049, Synthetic substances with œstrogenic activity. 1059, Naphthyl derivatives of barbituric acid. 1061, Alkaloid cuprichlorides. Alkaloids (various). New derivatives of p-arsanilic acid. 1063, Determination of novocaine and anæsthesin. 1064, Determining traces of nicotine. Identification of N-oxides of alkaloids. Colour reactions of alkaloids. 1077, Action of ultra-violet rays on alkaloid solutions. Pharmacology of substances from yeast. 1084, Prep. of purified bacteriophage.



## PATENTS.

[Manufacture of] ephedrine salts and oil solutions thereof. E. E. MOORE and E. B. CARTER, Assrs. to ABBOTT LABORATORIES (U.S.P. 1,861,543, 7.6.32. Appl., 29.3.30).—Ephedrine salts of fatty acids  $> C_8$  (e.g., erucic, oleic, ricinoleic) are sol. in mineral oil. C. H.

[Manufacture of] *d*-5-phenyl-5-ethylhydantoin [hypnotics]. H. SOBOTKA (U.S.P. 1,861,458, 7.6.32. Appl., 8.9.30).—The *d*-compound, m.p.  $235^\circ$ ,  $[\alpha]_D +118^\circ$  to  $+123^\circ$ , is free from the rash-producing property of the *l*- and *dl*-compounds. It may be obtained by resolution of the *dl*-compound with brucine or by synthesis from *d*- $\alpha$ -amino- $\alpha$ -phenylbutyric acid, amide, or nitrile, or from *d*- $\alpha$ -cyano- $\alpha$ -phenylbutyramide. C. H.

Manufacture of CC-[5:5]-phenylethyl-*N-n*-propylbarbituric acid. F. HOFFMANN-LA ROCHE & Co. A.-G. (B.P. 398,132, 5.5.33. Ger., 30.6.32).—5:5-Phenylethyl-*N*-allylbarbituric acid is reduced, e.g., with  $H_2$  and  $PdCl_2$  or Ni, to the *N*-Pr<sup>a</sup> compound, m.p.  $77-78^\circ$ , which is effective against epilepsy and less toxic than the *N*-Me or -Et compounds. (Cf. B.P. 391,741; B., 1933, 812.) C. H.

Manufacture of 3:4:5-trisubstituted 1:2:4-triazoles. SCHERING-KAHLBAUM A.-G. (B.P. 396,778, 30.1.33. Ger., 13.2.32).—3:5-Substituted furodiazoles are condensed with amines, in a solvent (EtOH) if desired. Triazoles are thus obtained from: 3:5-dimethylfurodiazole with  $NH_2Me$  (m.p.  $94.5^\circ$ ),  $NH_2Ph$  (m.p.  $235^\circ$ ), *p*- $NH_2 \cdot C_6H_4 \cdot OEt$  (m.p.  $161.5^\circ$ ), or 4-amino-2-butoxypyridine (m.p.  $154.5^\circ$ ); 5-phenyl-3-methylfurodiazole with  $NH_2Me$  (m.p.  $137^\circ$ ) or  $NH_2Ph$  (m.p.  $161^\circ$ ). C. H.

Manufacture of complex double compounds of organic heavy-metal mercapto [thiol] compounds. SCHERING-KAHLBAUM A.-G. (B.P. 398,020, 19.8.32. Ger., 7.11.31).—Heavy-metal compounds of thiocarbohydrates are treated with inorg. or org. SH compounds, other than thiocarbohydrates, in aq. or org. media. Examples include: Au thioglucose with  $Na_2S_2O_3$ , cysteine, or 3-thiolsulphanilic acid; Na 3-aurothiolsulphanilate with Na thioglucose or  $Na_2S_2O_3$ ; Sb thiocellobiose with Na thiolactate; Na cuprothiolactate with  $Na_2S_2O_3$ . C. H.

[Manufacture of] hydroxymercuri-derivatives of resorcin[ol] iodinated sulphonphthaleins. F. DUNNING (U.S.P. 1,863,268, 14.6.32. Appl., 23.2.29).—*o*-Sulphobenzoic acid is di- or tetra-iodinated, condensed with resorcinol, and mercured to give antiseptics and germicides. C. H.

Germicides. Phenylaminoalkylcarbinols. Conservation of pharmaceutical preps.—See III. Milk products.—See XIX.

## XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Photo-activity of dichromated colloids. H. M. CARTWRIGHT and H. MURRELL (Nature, 1933, 132, 603—604).—Addition of alkali to the dichromate bath of the gelatin-coated paper increases the contrast of the image by lowering the density of the weaker tones. This increase is interpreted in terms of the photo-activity of  $HCrO_4'$  and  $CrO_4''$ . L. S. T.

Recent developments in infra-red photography. O. F. BLOCH (Chem. & Ind., 1933, 829—837).

[Uses of] cellulose derivatives.—See V.

See also A., Oct., 1019, Formation of Ag in the photographic layer by X-rays. 1026, High-temp. X-ray camera. Determination of colour-sensitivity of photographic layers.

## PATENTS.

[Manufacture of] photographic emulsions [and sensitising dyes]. L. G. S. BROOKER, Assr. to EASTMAN KODAK Co. (U.S.P. 1,861,836, 7.6.32. Appl., 19.3.30).—Thioiso- and thio- $\psi$ -cyanines, obtained by condensing a methylnaphthathiazole alkiodide etc. with a quinoline alkiodide, are used as photosensitisers. C. H.

Photographic printing method. K. C. D. HICKMAN, Assr. to EASTMAN KODAK Co. (U.S.P. 1,897,844, 14.2.33. Appl., 11.2.31).—A sheet of a cellulose derivative (e.g., acetate or nitrate) is softened by applying a solvent to the surface, and the layer is then pressed into firm contact with the negative to be copied. The Ag grains in the surface of the negative impart a matt surface, and when the cellulose layer is removed after a few min. it will bear an accurate impression of those matt areas. The layer is moistened with glycerin and  $H_2O$  (1:1), or with gum arabic,  $H_3PO_4$ , and  $H_2O$  (25:1:100). After thorough wiping, the layer remains differentially selective to printing ink, and can be used as a printing plate. J. L.

## XXII.—EXPLOSIVES; MATCHES.

[Silver] azide detonator. F. BLECHTA (Chim. et Ind., 1933, 29, Spec. No., 921—925).— $AgN_3$  (I) is non-toxic, resistant to moist  $CO_2$ , and has a low min. charge (0.02 g. for tetryl); its disadvantages are its cost and colloidal nature. Admixture with inert cryst. substances would increase its min. charge. This may be reduced by mixing (I) with other explosive materials, such as tetryl, penthrite, and Hg fulminate. The mixtures are not liable to become dead-pressed; their brisance, in fact, increases with pressure. (I) is more stable than  $PbN_6$  and can be used in Cu or brass shells, being therefore suitable for use in fiery mines. The addition of the explosive substances facilitates ignition, thereby avoiding the need for adding Pb styphnate. Azides of Hg and Th have a higher min. charge than has (I). W. J. W.

Minimum charge of mercury fulminate. J. BARCİKOWSKI and J. KIELCZEWSKI (Chim. et Ind., 1933, 29, Spec. No., 939—944).—It is possible to reduce the min. charge by compressing the fulminate into a particular form, the one investigated representing two superimposed cylinders of various relative diam. and heights. Under suitable conditions of compression and confinement a charge of 0.1 g. will detonate trinitrotoluene. W. J. W.

Formation of nitrodiazotoluenesulphonic acid in trinitrotoluene wash-waters by the action of sodium sulphite. B. BATÍK (Chim. et Ind., 1933, 29, Spec. No., 960—963; cf. B., 1924, 451).—The wash-waters (I) contain dinitrotoluenesulphonates,  $NaNO_2$ , and excess of  $Na_2SO_3$ . On acidulation, sulphonates become reduced by the  $NaHSO_3$  and  $SO_3$  formed and



give a nitroaminosulphonic acid, which with excess of acid gives nitrodiazotoluenesulphonic acid, as well as diazoamino-compounds. These ignite readily and are susceptible to shock. Contact of (I) with mineral acids must therefore be avoided. The presence of these substances may serve to indicate asymmetrical isomerides in  $C_6H_2Me(NO_2)_3$ . W. J. W.

**Purification of trinitrotoluene by sodium sulphite; action on  $\alpha$ -trinitrotoluene.** J. MEČÍŘ (Chim. et Ind., 1933, 29, Spec. No., 952–959).—Factors in the purification are the relative proportions of  $C_6H_2Me(NO_2)_3$  and  $Na_2SO_3$ , and the temp. and duration of reaction. Mixtures of 2 : 4 : 6- $C_6H_2Me(NO_2)_3$  (I) and 2–6% of  $\alpha$ -trinitrotoluenes (II) require treatment for 3 hr. with a 5% solution at normal temp. for complete elimination of (II), the loss of (I) being 1.5%. At 50° the duration of the reaction is only  $\frac{1}{2}$  hr., but the loss is 5%.  $\alpha$ -Dinitrotoluenes react with sulphite at 50°, 50% being attacked in  $\frac{1}{2}$  hr., but in the cold the reaction proceeds more slowly. The 2 : 4-compound is not appreciably affected. Almost pure (I) (m.p. > 80.5°) can be obtained from mixtures containing 2 : 4- $C_6H_3Me(NO_2)_2$  and (II). The reaction between (I) and  $Na_2SO_3$  is analogous to that between (I) and alkalis. Hexanitrodibenzyl has been isolated as a final product. W. J. W.

**New method of initiation [of explosives].** A. MAJRICH (Chim. et Ind., 1933, 29, Spec. No., 926–932).—Initiation by basic Pb picrate (I), Pb trinitroresorcinate, Pb nitrate hypophosphite, Hg acetylide, and Na fulminate differs from that by other initiating compounds in that the former series burn extremely rapidly without detonation. This gives rise to adiabatic compression, which causes explosion of the secondary charge (S). This type of initiation may be termed explosion isomerism. Its effect depends on low compression of the primary charge (P). The combustion velocity of (I) decreases from 1780 to 431 m./sec. for increasing charge densities of 0.64–2.47 g./c.c. In applying the method to the manufacture of detonators, S (penthrite) is pressed into the base of the shell and covered with Cu foil, and P (enclosed in a capsule) is inserted into the shell so that there is an air space between it and S. W. J. W.

**Internal ballistics of smokeless powders as a liaison factor between manufacture and consumption.** V. BENDA (Chim. et Ind., 1933, 29, Spec. No., 1006–1010).—A study of internal ballistics serves to determine the grain size of powders required to give a desired result with a given weapon, and enables the wt. of charge within defined pressures to be prescribed. The development of, and modern methods of calculating, internal ballistics are outlined and illustrated by examples. W. J. W.

**Speed of combustion of two colloidal powders of different composition but the same explosion temperature.** H. MURAOUR and G. AUNIS (Bull. Soc. chim., 1933 [iv], 53, 613–617).—Equal temp. of explosion lead to equal rates of combustion in spite of differences in composition. Max. pressures were different but in agreement with the calc. vals. A. S. C. L.

**Testing of safety fuse with a galvanometer.** K. PEŠEK (Chim. et Ind., 1933, 29, Spec. No., 933–938).—

A battery of 300 volts max. and a mirror galvanometer are employed, the fuse being brought into the circuit by means of two metallic probes inserted into its powder core. Comparison is made with a standard (1 m.) length of fuse, which gives a scale deflexion of 0–50 with 40 volts. The equiv. voltages required by fuses of different counters to give an equal deflexion and the effect of coil diam. on deflexion are shown. Only graphited fuse can be tested by this method. W. J. W.

**Electrochemical stability test for explosives.** A. PAVLIK (Chim. et Ind., 1933, 29, Spec. No., 978–1005).—The Hansen test has been investigated and curves are given for nitroglycerin, nitrocellulose, smokeless powders, dynamite,  $C_6H_2Me(NO_2)_3$ ,  $C_6H_2(NO_2)_3 \cdot OH$ , penthrites, and  $T_4$  (trimethylenetrinitroamine). The quinhydrone electrode was used, as the H electrode has certain disadvantages, but even the former could only be used at  $p_H$  2–8, and the test rests on the assumption that  $HNO_2$  and  $HNO_3$  are completely dissociated in the test solution. The suitability of the test for various types of explosive is discussed, and the influence of the physical condition of the explosive, of stabilisers, humidity, and the presence, or removal by aspiration, of the decomp. gases, on the decomp. curves is shown. Heyrovsky's method with the dropping Hg cathode was examined. W. J. W.

**Preparation and testing of methanites.** V. PELANT (Chim. et Ind., 1933, 29, Spec. No., 888–901).—An improved method for testing the security (S) of methanites (I) by determination of the % of ignitions produced in galleries containing a known proportion of  $CH_4$  by repeated firing under standard conditions is suggested. The effects on S of the amounts of  $H_2O$ , various org. compounds, and of dissolved air on the dynamite are described, and the prep. of (I) of high S is discussed. A. A. L.

**Tests of safety explosives [methanite].** J. SEDIVÝ (Chim. et Ind., 1933, 29, Spec. No., 976–977).—Detonation velocity tests by Dautriche's method on whole charges and charges divided into two portions, respectively, gave the following results: Dynamite-I [using melinite cordeau (A)] 6290, 5980; [penthrite cordeau (B)] 6380, 5910 m./sec. With methanite (A; 4 samples) 2280, 950; 2120, 1190; 2010, 1005; 1630, 820; (B) 2380, 970 m./sec. The results partly explain why, in Germany, where tests are carried out with heavier charges, as used in the mines, greater safety is indicated. W. J. W.

**Determination of  $C_6H_4Me \cdot NO_2$ .**—See III. Metals in explosive manufacture.—See X.

See also A., Oct., 1017, Thermal decomp. of Me azide, and of 2 : 4 : 6-trinitro-1 : 3 : 5-triazobenzene. 1020, Action of  $\alpha$ -rays on explosives. 1038, Nitration of cellulose.

#### PATENTS.

**[Low-density] explosive composition.** R. SCHLESS, ASST. to PENNSYLVANIA POWDER CO. (U.S.P. 1,901,126, 14.3.33. Appl., 29.10.30).—The absorbent carbonaceous material consists of comminuted wheat straw, a convenient form of which is "Inso board" sawdust. The absorbent may receive pretreatment with  $NH_4NO_3$  or  $NaNO_3$ . W. J. W.



**Manufacture of explosive substance.** L. E. D'A. CALLERY (B.P. 397,600, 20.4.32).—Anhyd.  $\text{NH}_3$  gas is caused to bubble into a cooling tank containing 40% aq.  $\text{CH}_2\text{O}$  solution.  $\text{NH}_4\text{NO}_3$  is dissolved in the  $(\text{CH}_2)_6\text{N}_4$  produced, and the product is oxidised at  $15^\circ$  with  $\text{HNO}_3$  and  $\text{H}_2\text{O}_2$ . The pptd. crystals are dried in a centrifuge. W. J. W.

**Manufacture of nitrocellulose powders.** A. S. O'NEIL, Assr. to WESTERN CARTRIDGE Co. (U.S.P. 1,896,642, 7.2.33. Appl., 11.4.27).—In pulping purified nitrocellulose, the time of the operation and the adjustment of the beater knives are controlled so as to convert part of the material into hydrocellulose. Raw cotton may be added during the pulping. After addition of moderants and stabilisers, the pulped material is formed into sheets on a paper-making machine and subsequently dried and calendered. Multiple sheets may be produced, the outer layers having a low, and the middle layers a high, N content, a progressively burning powder being thereby obtained. W. J. W.

**Dehydrating nitrocellulose.**—See V.

### XXIII.—SANITATION; WATER PURIFICATION.

**Disinfection.** D. BACH (Bull. Sci. pharmacol., 1932, 39, 499—504; Chem. Zentr., 1933, i, 2124).—The disinfecting effect of  $\text{Pr}^+\text{CO}_2\text{H}$  on various bacteria is, in general, due to the non-ionised mol.; the average effect is  $>$  that of  $\text{EtCO}_2\text{H}$ . The disinfecting action of fatty acids is parallel with their surface activity. A. A. E.

**Disinfectants. II. Monobasic fatty acids.** D. BACH (Bull. Sci. pharmacol., 1932, 39, 425—434; Chem. Zentr., 1933, i, 2124).—Towards  $\text{HCO}_2\text{H}$  Gram-positive are more resistant than Gram-negative bacteria; usually 20—30 mg. per litre are sufficient, and the effect is generally due to the non-ionised mol. The disinfecting action of  $\text{AcOH}$  is 5% of that of  $\text{HCO}_2\text{H}$ ; that of  $\text{EtCO}_2\text{H}$  is  $>$  that of  $\text{AcOH}$ . A. A. E.

**Chemical control of the fouling of air by carbon dioxide.** F. P. E. CHAIGNON (Chim. et Ind., 1933, 29, Spec. No., 266—267).—A simple (modified Orsat) apparatus is described for determining  $\text{CO}_2$  contents up to about 2% in air. J. W. S.

**Lead poisoning from paints removed by sandblasting.** W. STOLDT (Pharm. Zentr., 1933, 74, 545—546).—Pb-poisoning of some cows was traced to the presence on the grass enclosure of Pb compounds which had their source in the red-Pb undercoat (removed by sandblasting) of a neighbouring bridge. S. M.

**Treatment and utilisation of [sewage] sludge.** F. C. VOKES (Engineering, 1933, 136, 317—318).—The application of the sludge-digestion process at Birmingham is described. C. J.

**Bio-aëration or activated sludge.** J. HAWORTH (Engineering, 1933, 136, 284—286).—Past and present methods of sewage purification are discussed with special reference to the activated-sludge process. A new bio-aëration plant is described in which the mixed liquor is circulated through the plant by a pump, the floor of the channels being graded to a suitable fall to enable the required lift to be given in one stage. Fixed baffles across the middle of the tank are introduced to disturb

the surface and so increase aëration and to adjust the static head to requirements. C. J.

**Oligodynamic action of metals.** K. KOHN and F. GRUSCHKA (Chim. et Ind., 1933, 29, Spec. No., 604—605).—Evidence is given in support of the theory that the bactericidal action of Cu, Ag, and Hg is due to the entry of ions of these metals into the aq. medium containing the bacteria. A. R. P.

**Use of copper and silver for the elimination of micro- and macro-organisms from swimming-bath water.** H. IVEKOVIC (Z. Gesundheitstsch., 1932, 24, 251—254, 314—320; Chem. Zentr., 1933, i, 2152—2153).

**Determination of water pollution by a biological reaction.** L. LLOYD (J. Hyg., 1933, 33, 183—195).—A small amount of a culture of aërotactic organisms, especially *Polytoma uvella*, is placed in a narrow tube and a column of  $\text{H}_2\text{O}$  is superimposed. The organisms aggregate in a blanket-like formation which is inversely proportional to the amount of dissolved  $\text{O}_2$ . The presence of  $\text{NO}_3^-$  or  $\text{NO}_2^-$  decreases the rate of climb, which is increased by organisms having an affinity for  $\text{O}_2$ . CH. ABS.

**Some methods in oilfield water analysis.** W. A. MACFADYEN (J. Inst. Petroleum Tech., 1933, 19, 679—686).—Details are given for the determination of  $\text{Cl}^-$ ,  $\text{SO}_3^{2-}$ ,  $\text{CaO}$ ,  $\text{Fe}^{++}$ , alkaline polysulphides, and  $\text{pH}$ . D. K. M.

**Trade wastes from gasworks.** A. McDONALD (Gas World, 1933, 99, 246—250).—The various means of disposing of gas liquor are discussed. Complete destruction by coke-quenching or in the producers or waste-gas flues leads to atm. pollution. Dilution of raw liquor which is then passed to the sewage works must be carried out in a regular flow to avoid excessive  $\text{O}_2$ -adsorption vals. Dephenolisation by solvent extraction and bacterial treatment of sulphate-works effluent is expensive. Economic results have, however, been obtained by use of a conc. gas-liquor plant, which also reduces the toxicity of the effluent. R. N. B.

**Determining dissolved  $\text{O}_2$  in  $\text{H}_2\text{O}$ .**—See I.  
**Determining  $\text{O}_2$  in waters.**—See VII.  
**Corrosion in  $\text{H}_2\text{O}$ -pipes.**—See X.

### PATENTS.

**[Cyanide] fumigation composition and process.** K. F. COOPER, Assr. to AMER. CYANAMID Co. (U.S.P. 1,889,950, 6.12.32. Appl., 2.4.30).—Claim is made for a finely-powdered mixture of  $\text{Ca}(\text{CN})_2$  and S to which may be added toxic or inert diluents. When used as a dusting powder for insecticidal purposes the mixture generates HCN by the action of the  $\text{O}_2$  in the air and a certain amount of  $\text{Cu}(\text{CNS})_2$  is also formed. A. R. P.

**Purification of water.** J. KRÜGER (B.P. 399,402; 7.3.33).—Hard  $\text{H}_2\text{O}$  is sprayed in a closed container on to bodies having a surface of  $\text{CaCO}_3$ , and flows over the material in countercurrent to a supply of steam so regulated that the  $\text{H}_2\text{O}$  is heated to the b.p., but all the steam is condensed and only expelled gases escape.

L. A. C.  
**Supplying air etc. to liquids.**—See I.  
**Germicides.**—See III.